1990

PERFORMANCE REPORT

WATER QUALITY SECTION

TD 380 P47 MOE



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1990 PERFORMANCE REPORT WATER QUALITY SECTION

Susan Janhurst

Laboratory Services Branch
Ontario Ministry of the Environment

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INTRODUCTION

The Water Quality Section is part of the Ministry of the Environment's Laboratory Services Branch (LSB). The Section provides the Ministry with expertise in inorganic chemistry and microbiology. The largest number of tests in the branch are handled by the Water Quality Units, where staff analyze a broad spectrum of environmental sample types including: ground water, surface water, drinking water, precipitation, sewage, industrial waste, leachate, soil and soil extract.

This report provides an outline of the section's quality control (QC) programs for inorganic chemistry and microbiology, along with a summary of the resulting 1990 performance data for each test. The Water Quality Section strives to maintain a high standard of analytical performance through its quality assurance program and QC is an integral part of the process.

ACKNOWLEDGEMENTS

This report is dedicated to the technicians of the Water Quality Section.

and

The author would like to thank the technical staff of the Water Quality Section for their assistance in accumulating the quality control data, and gratefully acknowledges the contribution provided by all supervisors; Louis Dubreuil for conducting preliminary statistical analysis on the quality control data collected; Marion Stuart for assisting in updating the test descriptions, data summaries and graphics pages.

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1.0 PERFORMANCE REPORT FORMAT

The performance report is divided into three parts. Part One outlines the format of this report, Parts Two and Three consist of quality control programs and performance summaries for chemistry and microbiology respectively.

A performance report is generated for each test conducted in the Water Quality Section with the exception of those parameters where no data or less than three pieces of data exist for 1990. The performance report is set up accordingly: first by the alphabetical name of the test eg. Total Organic Carbon is filed under the heading "Carbon, Total Organic"; second by the work station code; and third by the test name code. Where more than one test is performed at a work station, then the test name is bracketed in the title. Each performance report usually consists of three pages: the test description page, the performance data summary page, and the quality control graphics page. The quality control graphics page may not be included for those parameters that do not use quality control standards, or where less than ten pieces of Calibration Control data exist. Detailed information concerning each of these pages is outlined next.

1.1 TEST DESCRIPTION PAGE

TITLE:

The name of the test parameter.

IDENTIFICATION:

Laboratory:

Location where the test is performed.

LIS* Test Name Code:

LIS code for analysis request.

Work Station Code:

LIS code for sample routing to the work station.

Method Code:

LIS code for the analytical procedure.

Sample Type/Matrix:

The various sample types that can be routed to

the work station.

Method Introduced:

Date that the method was implemented at the laboratory.

Units:

Unit of measurement in which the results are reported.

Unit Code:

LIS code for the unit of measurement in which the

results are reported.

Supervisor:

Name of supervisor responsible for the designated laboratory.

^{*}LIS - Laboratory Information System

SAMPLING:

The type of container and preservative (if applicable) that is used and minimum volume of sample that is usually required (10). Any sample preparation that is normally performed in the field, is also indicated.

SAMPLE PREPARATION:

Sample preparation techniques which are usually performed at the laboratory before analysis.

ANALYTICAL PROCEDURE:

Analytical method used to determine the parameter.

INSTRUMENTATION:

Type of instrumentation, used to perform the test. Automated continuous flow systems, consist of a sampler, peristaltic pump, manifold for reagent addition, detection system and a readout system. Microcomputers are used to control the operation of analytical equipment and/or data acquisition.

REPORTING:

W and T are low level data qualifiers assigned to data that are near or below the detection limit values (3)(5). The code <W indicates that no measurable response was observed under the test conditions. The reported value indicates the smallest amount that could have been measured under routine conditions. W is smaller than the standard deviation of duplicates near zero. The <T code is used to represent a measurable amount of the analyte which under the test conditions is not verifiable. The reported result should be used only for large batches of similar data to evaluate background levels or trends of contaminants in the environment where more sensitive analytical methods are not available.

To provide a consistent Laboratory Services Branch approach to data reporting, the Water Quality Section calculates W from the standard deviation of duplicates (S_2) , near zero, by rounding down to the nearest 1,2 or 5 digit. T is five times W. The latest calculations, valid at date of publication for W and T values of all active work stations, are contained in this report. (APPENDIX B)

CALIBRATION:

The number of standards used to calibrate the analytical system plus blanks if applicable.

CONTROLS:

The calibration, drift, recovery, and interference controls that are used when applicable to ensure that the system is operating properly.

MODIFICATIONS:

Modifications to the test in 1991.

NOTES:

Explanatory notes which may aid the data user in interpreting results and information.

1.2 PERFORMANCE DATA SUMMARY PAGE

TITLE:

The name of the test parameter.

OUALITY CONTROL DATA FROM/TO:

The period of time during which data were collected.

LAB:

The laboratory in which the data were collected.

ANALYTICAL RANGE:

The full scale value for the analytical range is given in concentration units.

CALIBRATION CONTROL:

A table for the calibration control standards. The between run standard deviation (S), the within run standard deviation (S_w), the ratio S/S_w, and the ranges for acceptance limits of the control standards sums and differences.

RECOVERIES (Where applicable):

A table for the recovery control standards.

DUPLICATES:

A table of within run duplicate data. The data are sorted into a number of concentration spans. The coefficient of variation (%) is obtained by dividing the mean standard deviation (S_2) for a particular concentration span by the mean concentration of duplicate results in that span and multiplying by 100.

OTHER CHECKS (Where applicable):

A table for other checks.

1.3 QUALITY CONTROL GRAPHICS PAGE

TITLE:

The name of the test parameter.

DATE FROM/TO:

Period of time over which data were collected.

CALIBRATION CONTROL:

Calibration control standards sums and differences are plotted on a horizontal scale for the period of data collection (referred to on the graphs as "QUALITY CONTROL SAMPLE A+B" for example). The vertical scale consists of the control limits expressed on either side of the expected value. Control limits were chosen from previous analytical performance when available.

PART 2.0

CHEMISTRY

2.1 Quality Control Program, Chemistry

Quality control is a continuous process that involves constant checks of sample processing. Control activities that are conducted before sample analysis begins are checks on reagent chemicals, water purity, materials that are in contact with sample, and calibration.

Reagent chemicals are selected according to specific test method requirements.

Water purity is checked by daily monitoring for conductivity. Operations generally require conductivity levels of ≤ 1 uS/cm. Some procedures require purer water and this is accomplished by further refining the distilled water through a deionizing system.

Material checks are done on sample containers, filters, glassware and other equipment. These are checked for leaching, adsorption and contamination.

Calibration is conducted by analyzing a series of calibration standards covering the analytical range. Since a high degree of both precision and accuracy is required to detect and minimize any between-run changes, the standards are analyzed with as little handling as possible.

Once a system has been calibrated, quality control begins. Depending on the analytical procedure, quality control may be used to evaluate: calibration, blank, recovery, sensitivity, potential interference, and sample repeatability.

Calibration and Blank

Calibration is controlled by a minimum of two quality control standards and a long term blank which are prepared and maintained independently of the calibration standards. The system is not calibrated with the quality control standards. The long term blank is prepared identical to the quality control standards but with zero concentration of the analyte. Control standards are prepared less frequently than calibration standards and errors in newly prepared calibration standards can be detected by this cross check. Newly prepared control standards are run in parallel with the old control standards and must meet control requirements over three consecutive runs before the new standards are accepted on line.

The control standards data are assessed and compared against the control limits established from previous data to determine whether the calibration process is in control. The control limits are examined yearly and may be adjusted if the method performance improves and/or the historical data base is increased. Control limits are calculated for the sum (A+B) and differences (A-B) of the control standards by the equations $(A+B)\pm 4.0\times S_{A-B}$ and $(A-B)\pm 3.0\times S_{A-B}$ respectively (1). If a control limit is exceeded, the analysis is stopped, corrective action taken and the control standards are re-analyzed.

The standard deviation of the control standards is used to estimate the between run standard deviation (S) and is compared against the within run standard deviation (S_w). If the ratio S/S_w exceeds 1.5 then poor control of systematic error can be inferred (1). Values for S and S_w are calculated as follows:

$$2S^2 = (S_A)^2 + (S_B)^2$$
 $2S_w^2 = (S_{A-B})^2$

Where

 S_A = standard deviation of control standard A

 S_B = standard deviation of control standard B

 S_{A-B} = standard deviation of the difference between control standards A and B

NOTE: If a second range is employed for a test, more control standards are used because, in many systems, the between run standard deviations are concentration dependent.

Detailed description of the quality control processes are outlined in several LSB reports (2)(3)(4)(5).

Recovery

Some methods require sample pre-treatment, such as digestion or extraction. A recovery check, suitable to that method, is required to estimate the efficiency of the pre-treatment. Recovery standards are usually prepared at 0%, 20% and 80% of full scale. The solutions are analyzed in the same manner as routine samples. Although these solutions are not used to calibrate the instrument, corrections for the blank and matrix effects are calculated and applied if necessary. For an analytical run to be accepted, the recoveries should be within $\pm(5\% + T/2)$ of their expected values. (T is defined in Appendix A). The average blank should be within three standard deviations of its historical mean. If a second range is employed for a test, at least one additional recovery standard is used.

Sensitivity and Baseline

Any change in the sensitivity of the instrumentation is monitored periodically by analyzing a standard that is usually 80% of full scale, and comparing the peak height to the original calibration standards. Baseline drift is usually recorded by periodic analysis of deionized, distilled water (DDW) which does not contain any of the analyte, but may be adjusted to correspond to sample pre-treatment.

Interference

Interference checks are run on any test where a substance may be present in large enough concentration to affect the results. The checks are near the threshold concentration, beyond which the methodological safeguards used to minimize the interferences are no longer effective. These checks indicate that the interferences have no effect up to the specified concentrations. Spiked samples are not analyzed on a routine basis.

Sanmple Repeatability

Generally, one sample out of twenty is run in duplicate up to a maximum of three per day. The samples are selected for non-adjacent, within-run duplicate analyses. By analyzing samples in duplicate, the ability of the analyst to obtain repeatable analytical results, within an analytical run, can be determined. For results to be acceptable, at least two-thirds of the duplicate data must conform to limits which are based on historical performance.

The observed differences in duplicate results are accumulated and sorted according to sample concentration span. A standard deviation is calculated for each sample concentration span. The algorithm differs from the conventional standard deviation as follows:

Conventional Std. Dev.

Std. Dev. of Duplicates

(1) (2) standard deviations calculated for the performance report data summary pages

$$S_1 = \sqrt{\frac{\sum_{i=1}^{n} (\overline{x} - x_i)^2}{n-1}}$$

$$S_2 = \sqrt{\frac{\sum_{i=1}^{n'} (x_1 - x_2)_i^2}{2n'}}$$

Where

 S_1 = sample standard deviation

 $S_2 =$ duplicate difference standard deviation

n = number of data

 \overline{x} = mean of data

 $x_i = i^{th} \text{ result}$

 $(x_1 - x_2)_i$ = difference of the i^{th} duplicate

n' = number of duplicate pairs

Reported values for duplicate standard deviations have been treated by robust statistical methods (6)(7). The standard deviation (S_2) of the duplicate difference is also expressed as the coefficient of variation (CV)

$$CV = \frac{s_2}{\bar{r}} \times 100$$

2.2 PERFORMANCE SUMMARIES CHEMISTRY

*** ACIDITY ***

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code

Method Code

: Titration

: ACDT : PHACD

: 001BT2

Method Introduced

Units Unit Code : 01/05/79

: mg/L as CaCO₃

: 064915 Supervisor : F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow, Domestic Waters, Rivers, Lakes (by special

request: Industrial Waste, Sewage)

SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Sample aliquots (10.0 mL) are titrated in an automated system with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. pH and Gran acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration:

LTBL plus 2 standards, e.g. QCA

ACIDITY

QUALITY CONTROL DATA FROM 02/01/90 TO 12/12/90

Lab: Titration

Analytical Range: - to 100.0 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	105	25.0	24.92	-0.08	0.6567
B :	105	10.0	10.33	0.33	0.3790
A+B:	105	35.0	35.25	0.25	0.8894
A-B:	105	15.0	14.59	-0.41	0.5990

s.d.(AB) S(between runs): 0.54

Sw(within run): 0.42 S/Sw: 1.26

On any given day the calibration is accepted if the values obtained lie within the ranges:

32.57 - 37.43 for A+B 13.38 - 16.62 for A-B

DUPLICATES:

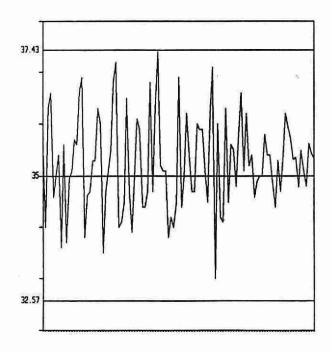
Number of Data Pairs	C	Sam oncn	ple Span	Mean(2) s.d.	Coefficient of var.(%)
50	0.0	-	2.0	0.1076	9.4
92	2.0		5.0	0.1234	3.8
40	5.0	-	25.0	0.1798	2.1
0	25.0		100.0	N.A	N.A
182	(Over	all	0.1307	

OTHER CHECKS:

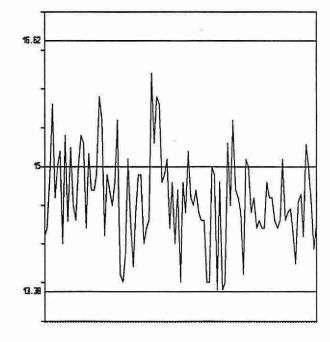
	Number	Data	Standard(1)
	of Data	Mean	Deviation

Long Term Blank	103	0.800	0.317

QUALITY CONTROL DATA FROM 02/01/90 TO 12/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** ACIDITY, GRAN ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: 01/08/82

LIS Test Name Code Work Station Code : ACDG : PHACD Units Unit Code : ug/L as H⁺ : 064801

Method Code

: 001BT5

Supervisor

: F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Sample aliquots (10.0 mL) are titrated with 0.01 N sodium hydroxide to pH >8.3. The titrant is standardized against 0.0005 N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. Data are subjected to Gran analysis.

pH and total fixed endpoint acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration: LTBL (expected result is 16.6 ug/L as H) plus 2 standards, e.g. QCA

ACIDITY, GRAN

QUALITY CONTROL DATA FROM 02/01/90 TO 12/12/90

Lab: Titration

Analytical Range: - to 1000 ug/L as H+

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			****************	*********	**********
A :	105	500.0	498	-2.0	12.92
B :	105	200.0	205	5.0	7.38
A+B:	105	700.0	702	2.0	17.57
A-B:	105	300.0	293	-7.0	11.58

s.d.(AB) S(between runs): 10.5

Sw(within run): 8.19 S/Sw: 1.28

On any given day the calibration is accepted if the values obtained lie within the ranges:

657.7 271.8

742.3 328.2 for A+B for A-B

DUPLICATES:

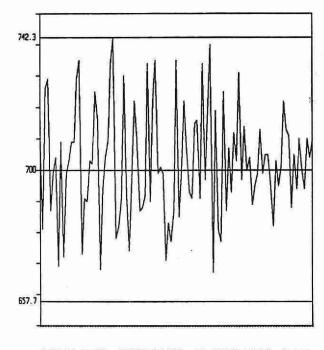
Number of Data Pairs	*	Samp Concn		Mean(2) s.d.	Coefficient of var.(%)
*******				**********	
52	0		40	2.017	1.9
95	40		100	2.924	2.9
38	100		500	2.983	2.0
0	500		1000	N.A.	N.A.
185		Overal	1	2.699	magnista ji

OTHER CHECKS:

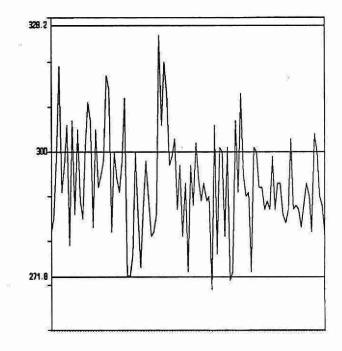
	Number of Data	Data Mean	Standard(1) Deviation
8:			
Long Term Blank	103	13.72	4 128

ACIDITY, GRAN (ug/L as H')

QUALITY CONTROL DATA FROM 02/01/90 TO 12/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** ALKALINITY ***

IDENTIFICATION:

Laboratory : Dorset

Method Introduced : 26/07/79 LIS Test Name Code : ALKT Units : mg/L as CaCO₃

Work Station Code : DOT : 064915 Unit Code Method Code : 0905T3 Supervisor : A. Neary

Sample Type/Matrix : Streams, Lakes, Precipitation, Groundwaters

SAMPLING:

Quantity Required : 150 mL

Container : Plastic

ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.05 T value: 0.25

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration : LTBL plus 2 standards, e.g. QCA

Drift : 2 standard buffers - 2 times daily

ALKALINITY

QUALITY CONTROL DATA FROM 04/01/90 TO 14/12/90

Lab: Dorset

Analytical Range: - to 80.00 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	209	20	19.98	-0.02	0.308
B :	209	5	4.89	-0.11	0.121
A+B:	209	25	24.88	-0.12	0.394
A-B:	209	15	15.09	0.09	0.252

s.d.(AB) S(between runs): 0.23 Sw(within run): 0.18 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.28 26.72 for A+B 13.50 16.50 for A-B

DUPLICATES:

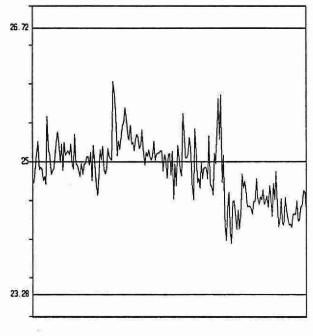
Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
********				*******	
279	0.00	-	5.00	0.092	4.6
125	5.00	4	10.00	0.114	2.2
63	10.00		80.00	0.131	1.1
467	(Overall		0.103	

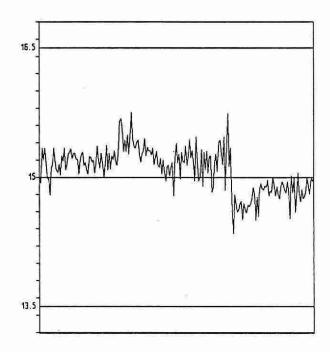
OTHER CHECKS:

	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	209	1.64	0.258

ALKALINITY (mg/L as CaCO3)

QUALITY CONTROL DATA FROM 04/01/90 TO 14/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** ALKALINITY ***

IDENTIFICATION:

Laboratory

Method Code

: Titration

LIS Test Name Code Work Station Code

: ALKT : RATS

: 004AT6

Sample Type/Matrix

: Rivers, Lakes, Precipitation

Method Introduced

Units

: 09/07/80

: mg/L as CaCO₃

Unit Code : 064915 Supervisor : F. Lo

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH <4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. pH, Gran alkalinity, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration

: BL plus 4 standards, e.g. QCA

Drift

: In run standards throughout the run (tap water diluted to 20% V/V)

ALKALINITY

QUALITY CONTROL DATA FROM 03/01/90 TO 27/12/90

Lab: Titration

Analytical Range: - to 1000 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	*******		***************************************		************
A :	78	250.0	249.6	-0.4	1.672
B :	78	50.0	48.2	-1.8	1.338
A+B:	78	300.0	297.9	-2.1	2.279
A-B:	78	200.0	201.4	1.4	1.993
C :	78	10.0	10.09	0.09	0.288
D:	78	2.5	2.53	0.03	0.127
C+D:	78	12.5	12.62	0.12	0.350
C-D:	78	7.5	7.55	0.05	0.275

s.d.(AB) S(between runs): 1.51

Sw(within run): 1.41 S/Sw: 1.1

s.d.(CD) S(between runs): 0.22

Sw(within run): 0.19 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

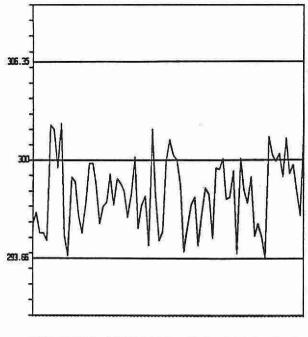
293.66 306.35 for A+B 195.77 204.23 for A-B 13.33 11.67 for C+D 6.95 8.05 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
47	0.0		50.0	0.3606	2.4
26	50.0		100.0	0.7350	ĩ.i
142	100.0		350.0	1.6159	0.8
0	350.0		1000.0	N.A	N.A
215		Overa	il	1 2431	

ALKALINITY (mg/L as CaCO3)

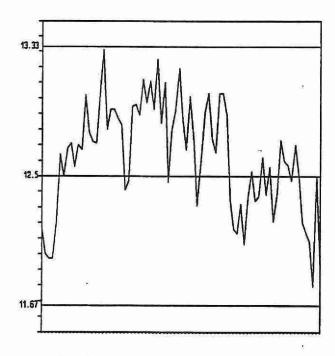
QUALITY CONTROL DATA FROM 03/01/90 TO 27/12/90



204.23

QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B



7.5

QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D
CONTROL LIMIT

*** ALKALINITY ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: 09/07/80

LIS Test Name Code

: ALKT : WATS Units Unit Code : mg/L as CaCO₃

Work Station Code Method Code

: WATS

Supervisor

: 064915 : F. Lo

Sample Type/Matrix

: Domestic Waters, Sewage, Effluents

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

pH, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration

: BL plus 3 standards, e.g. QCA

Drift

: In run standards throughout the run (tap water diluted to 50% V/V)

ALKALINITY

QUALITY CONTROL DATA FROM 02/01/90 TO 29/11/90

Lab: Titration

Analytical Range: - to 1000 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	******	*******	*********	*******	
A :	152	250	249.3	-0.7	1.912
В:	152	100	100.2	0.2	1.328
A+B:	152	350	349.5	-0.5	2.720
A-B:	152	150	149.1	-0.9	1.856
C :	152	100	99.2	-0.8	0.805
D:	152	25	24.7	-0.3	0.296
C+D:	152	125	124.0	-1.0	0.938
C-D :	152	75	74.5	-0.5	0.770

s.d.(AB) S(between runs): 1.65 Sw(within run): 1.31 S/Sw: 1.25

s.d.(CD) S(between runs): 0.61 Sw(within run): 0.54 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

341.23 358.78 for A+B 144.15 155.85 for A-B 119.20 130.80 for C+D 71.13 78.87 C-D for

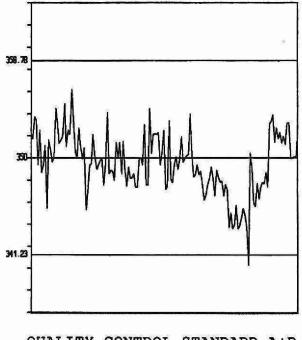
DUPLICATES:

Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
*********	****			********	
26	0.0	-	25.0	0.3951	5.3
114	25.0	-	100.0	1.0371	1.8
207	100.0	•	500.0	2.5452	2.4
6	500.0	-	1000.0	3.0779	1.3
353	9	Overa	dl l	1.8941	

ALKALINITY (mg/L as CaCO3)

155.85

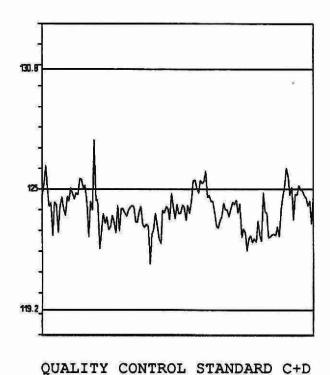
QUALITY CONTROL DATA FROM 02/01/90 TO 29/11/90

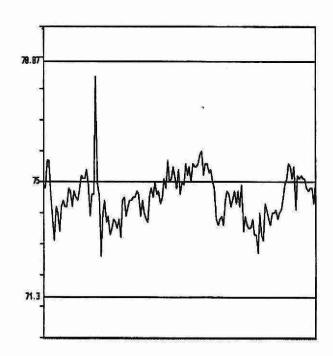


14.15 14.15

QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B





TANDAND CID

QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

*** ALKALINITY ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: Before 1980

LIS Test Name Code

: ALKT : WOSDIRT Units

: mg/L as CaCO₃

Work Station Code Method Code

: WQSDIR1

Unit Code Supervisor : 064915 : F. Lo

Sample Type/Matrix

: Landfill leachates

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples are pipetted manually (50.0 mL) and titrated with 0.02 N sulphuric acid to pH endpoint of 4.5. Analysis is performed on the supernatant or filtrate.

INSTRUMENTATION:

Automated modular titration system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration

: BL plus 2 standards, e.g. QCA

ALKALINITY

QUALITY CONTROL DATA FROM 08/01/90 TO 17/12/90

Lab: Titration

Analytical Range: - to 1000 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	37	570.0	571.6	1.6	2.991
B :	37	114.0	116.3	2.3	1.389
A+B:	37	684.0	688.0	4.0	3.520
A-B:	37	456.0	455.3	-0.7	3.059

s.d.(AB) S(between runs): 2.33

Sw(within run): 2.16 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

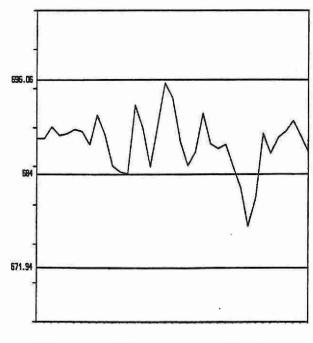
671.94 - 696.06 for A+B 447.96 - 464.04 for A-B

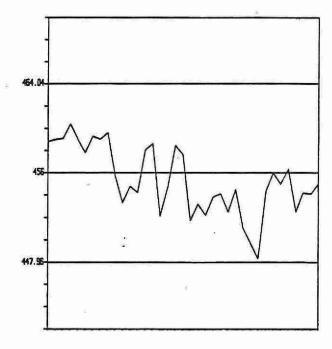
DUPLICATES:

Number of Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
20	0.0 - 150.0	0.566	1.2
17	150.0 - 250.0	1.653	4.4
16	250.0 - 500.0	3.014	3.7
7	500.0 - 1000.0	15.558	2.9
60	Overall	1.700	

ALKALINITY (mg/L as CaCO3)

QUALITY CONTROL DATA FROM 08/01/90 TO 17/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

*** ALKALINITY ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 21/10/85

LIS Test Name Code

: ALKT3

Units

: mg/L as CaCO₃

Work Station Code Method Code

: 0905T3

Unit Code Supervisor : 064915 : A. Neary

Sample Type/Matrix

: Streams, Lakes, Precipitation, Groundwaters

SAMPLING:

Quantity Required

:150 mL

Container

: Amber polyethylene bottle filled to the brim; screw cap with cone-shaped liner is

preferred.

ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 3.8. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 2 standard buffers - once daily

ALKALINITY

QUALITY CONTROL DATA FROM 04/01/90 TO 14/12/90

Lab: Dorset

Analytical Range: - to 100.0 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	209	20.00	20.16	0.16	0.525
B :	209	5.00	4.86	-0.14	0.364
A+B:	209	25.00	25.02	0.02	0.831
A-B:	209	15.00	15.29	0.29	0.352

s.d.(AB) S(between runs): 0.25 Sw(within run): 0.45 S/Sw: 1.8

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.28 26.72 for A+B 13.50 16.50 for A-B

DUPLICATES:

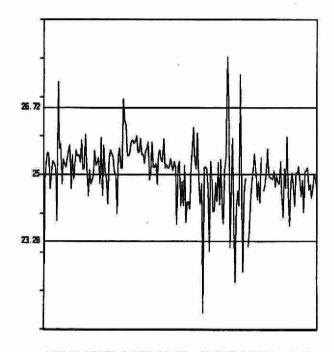
Number of Data Pairs	Sample Conen Span		Mean(2) s.d.	Coefficient of var.(%)

505	0.0	- 20.0	0.362	3.8
36	20.0	- 50.0	0.515	1.6
6	50.0	- 100.0	0.844	2.3
547	Ov	erall	0.373	

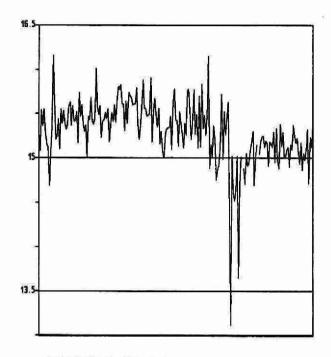
	Number of Data	30	Data Mean	Standard(1) Deviation
Long Term Blank	211	*	8.84	0.842

ALKALINITY (mg/L as CaCO3)

QUALITY CONTROL DATA FROM 04/01/90 TO 14/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

*** ALKALINITY, GRAN ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 26/07/79

LIS Test Name Code

: ALKTI

Units

: mg/L as CaCO₃

Work Station Code

: DOT

Unit Code

: 064915

Method Code

: 0905T6

Supervisor

: A. Neary

Sample Type/Matrix

: Streams, Lakes, Precipitation, Groundwaters

SAMPLING:

Quantity Required

:150 mL

Container

:250 mL Amber polyethylene bottle filled to the brim; screw caps with cone-shaped

liners are preferred.

ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH <3.7. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis. N.B. pH is determined simultaneously.

INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 2 standard buffers - 2 times daily

ALKALINITY, GRAN

QUALITY CONTROL DATA FROM 04/01/90 TO 14/12/90

Lab: Dorset

Analytical Range: - to 25.00 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
				*******	***************************************
A :	209	20.00	20.11	0.11	0.317
B :	209	5.00	4.98	-0.02	0.217
A+B:	209	25.00	25.09	0.09	0.465
A-B :	209	15.00	15.14	0.14	0.283

s.d.(AB) S(between runs): 0.27 Sw(within run): 0.20 S/Sw: 1.36

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.28 13.85 26.72 16.15 for A+B for A-B

DUPLICATES:

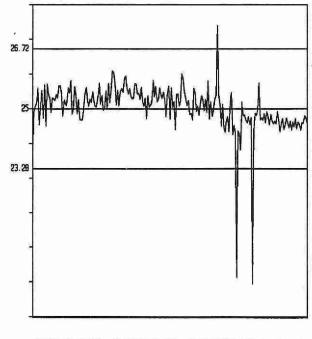
Number of Data Pairs	Control of the Contro		Mean(2) s.d.	Coefficient of var.(%)	

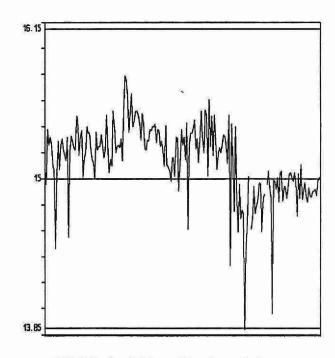
214	-13.0	-	2.0	0.123	N.A
78	2.0		5.0	0.114	3.9
44	5.0	-	10.0	0.137	2.9
23	10.0	-	25.0	0.180	1.3
359	(Overa	11:	0.126	

	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	211	-0.21	0.267

ALKALINITY, GRAN (mg/L as CaCO3)

QUALITY CONTROL DATA FROM 04/01/90 TO 14/12/90





QUALITY CONTROL STANDARD A+B

' QUALITY CONTROL STANDARD A-B

*** ALKALINITY, GRAN ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: 09/07/80

LIS Test Name Code Work Station Code : ALKTI : RATS Units
Unit Code

: mg/L as CaCO₃

Method Code

: 004AT6

Supervisor

: 064915 : F. Lo

Sample Type/Matrix

: Rivers, Lakes, Precipitation

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH <4.0. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis.

pH, total fixed endpoint alkalinity, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration

: BL plus two standards, e.g. QCA

Drift

: In run standards throughout the run (diluted tap water 20% V/V)

ALKALINITY, GRAN

QUALITY CONTROL DATA FROM 11/01/90 TO 11/12/90

Lab: Titration

Analytical Range: - to 25.0 mg/L as CaCO₃

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

C :	37	10.0	10.193	0.193	0.2311
D :	37	2.5	2.565	0.065	0.0874
C+D:	37	12.5	12.758	0.258	0.2564
C-D:	37	7.5	7.628	0.128	0.2372

s.d.(CD) S(between runs): 0.175 Sw(within run): 0.168 S/Sw: 1.04

On any given day the calibration is accepted if the values obtained lie within the ranges:

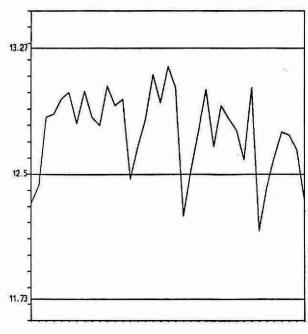
11.73 13.27 for C+D 6.99 8.01 for C-D

DUPLICATES:

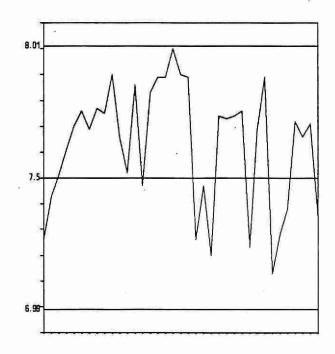
Number of Data Pairs	Sample Concn Span			Mean(2)	Coefficient
Data Fails			·	s.d.	of var.(%)
6	-2.0	= (1.0	0.1763	N.A
14	1.0	-	5.0	0.2597	7.8
8	5.0	•	25.0	0.4057	6.1
28		Overal	l	0.2955	

ALKALINITY, GRAN (mg/L as CaCO3)

QUALITY CONTROL DATA FROM 11/01/90 TO 11/12/90



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** ALUMINUM, ACID AMMONIUM OXALATE EXTRACTABLE ***

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 1986

LIS Test Name Code

: ALEOX

Units

: % by wt as Al

Work Station Code Method Code : DOMETOX : 302AA5 Unit Code

: 070813

Sample Type/Matrix

: Soil

Supervisor : A. Neary

SAMPLING:

Quantity Required

: 1 g

Container

: Glass or plastic

SAMPLE PREPARATION:

Samples are air-dried, disaggregated and sieved to less than 2 mm. A subsample is ground to <500 um (35 mesh).

ANALYTICAL PROCEDURE:

Samples (0.25 g) are weighed into disposable tubes. 10 mL of acid ammonium oxalate extractant is added and the tubes are capped and shaken for 4 hours in the dark. Samples are centrifuged and the analysis is performed on the supernatant. The solution is analyzed by AAS at 309.3 nm with a NO₂-acetylene flame. N.B. Iron, manganese and silicon may be determined on the same extract.

INSTRUMENTATION:

Varian AA 1275

REPORTING:

Maximum Significant Figures: 2

Current W value: 0.01

T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration

:Three long term soil samples, representing different soil types, 2 method blanks,

QC solutions at 25% and 75% of scale, round robin ECSS samples.

Drift

:BL plus 1 standard (100% F.S.) every 10 samples.

ALUMINUM, ACID AMMONIUM OXALATE EXTRACTABLE

QUALITY CONTROL DATA FROM 30/03/90 TO 14/12/90

Lab: Dorset Soils

Analytical Range: - to 2.00 % by wt. Al

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			***********	*********	************
A :	4	1.50	1.49 7	-0.003	0.054
В:	4	0.50	0.510	0.010	0.022
A+B:	4	2.00	2.007	0.007	0.071
A-B:	4	1.00	0.987	-0.013	0.041

s.d.(AB) S(between runs): 0.041

Sw(within run): 0.029 S/Sw: 1.41

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.70 - 2.30 for A+B 0.80 - 1.20 for A-B

RECOVERIES:

	Number of Data	Av. Concn Measured	Standard(1) Deviation
	**********		************
R1:	3	0.340	0.017
R2:	3	0.563	0.049
R3:	3	0.373	0.323

DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
	*****				*************
9	0.00	-	0.40	0.006	3.6
0	0.40	100	1.00	N.A	N.A
0	1.00		2.00	N.A	N.A
9	Overall			0.006	

	Number of Data	Data Mean	Standard(1) Deviation

Method Blank	4	0	0

ALUMINUM, CITRATE-BICARBONATE-DITHIONITE EXTRACTABLE

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: ALEDI : DOMETDI Units

: % by weight Al

Work Station Code

: 301AA5

Unit Code

: 070813

Method Code Sample Type/Matrix : Soil

Supervisor

: A. Neary

SAMPLING:

Quantity Required

 $0.5 \, \mathrm{g} \, \mathrm{dry}$

Container

Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2mm and a subsample ground to <500um (35 mesh)

ANALYTICAL PROCEDURE:

Aluminum is extracted from a 0.25 g soil sample using sodium citrate, sodium bicarbonate and sodium dithionite at 80°C (procedure is repeated twice). The sample is washed twice and its washings and extracts are combined and diluted to 50 mL with deionized water. The final solution is analyzed by AAS at 309.3 nm with a NO₂-acetylene flame.

Approximate absorbance: 0.3 at the full scale level

N.B. Iron (and Manganese, when required) may be determined on the same extract.

INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration:

Three soil samples representing different soil types; two QC solutions at 25% and 75% of

full scale, 2 method blanks; round robin ECSS samples (run occasionally).

Drift:

BL plus 1 standard (100% F.S.) every 10 samples

NOTES:

Values for recoveries are unknown - average value used.

ALUMINUM, CITRATE-BICARBONATE-DITHIONITE EXTRACTABLE

QUALITY CONTROL DATA FROM 05/04/90 TO 24/11/90

Lab: Dorset Soils

Analytical Range: - to 1.00 % by wt. Al

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	5	0.75	0.744	-0.006	0.0089
B :	5	0.25	0.248	-0.002	0.0109
A+B:	5	1.00	0.992	-0.008	0.0045
A-B:	5	0.50	0.496	-0.004	0.0194

s.d.(AB) S(between runs): 0.01

Sw(within run): 0.014 S/Sw: 0.73

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.92 - 1.07 for A+B 0.45 - 0.55 for A-B

RECOVERIES:

	Number	Av. Concn	Standard(1)
	of Data	Measured	Deviation
R1:	5	0.34	0.040
R2:	5	0.60	0.057
R3:	5	0.38	0.050

DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*********					*****************
8	0.00	-	0.20	0.0175	13.9
3	0.20	-	0.50	0.0081	2.9
3	0.50	-	1.00	0.0719	50.1
14	(Overall	-14	0.0191	

ā	Number of Data	Data Mean	Standard(1) Deviation
		***********	**********
Method Blank	5	0.000	0.0000

*** ALUMINUM, EXCHANGEABLE CATION ***

IDENTIFICATION:

Laboratory

: Dorset Soils

: 01/06/80

LIS Test Name Code

: ALESC

Method Introduced : meg/100 g Units

Work Station Code Method Code

: DOCATION : 306AA1

Unit Code : 355000 Supervisor : A. Neary

Sample Type/Matrix

: Soil

SAMPLING:

Quantity Required

: 6 g dry

Container

: Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for Al by AAS at 309.3 nm with a NO₂-acetylene

Approximate absorbance: 0.2 at the full scale level.

N.B. Calcium, magnesium, and potassium are determined on the same extract.

INSTRUMENTATION:

- -Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump
- -Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration

: Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of

full scall; 2 method blanks; round robin ECSS samples (run occasionally).

Drift

: BL plus 1 standard (100% F.S.) every 10 samples

NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown - average value used.

ALUMINUM, EXCHANGEABLE CATION

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90

Lab: Dorset Soils

Analytical Range: - to 2.50 meq/100 g

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		The second of the back of the			
A :	17	1.88	1.893	0.013	0.0429
B :	17	0.63	0.639	0.009	0.0225
A+B:	17	2.51	2.533	0.023	0.0493
A-B:	17	1.25	1.254	0.004	0.0474

s.d.(AB) S(between runs): 0.034

Sw(within run): 0.033 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.24 - 2.78 for A+B 1.06 - 1.44 for A-B

RECOVERIES:

	Number of Data	Av. Concn Measured		Standard(1) Deviation

R1:	17	1.503		0.0774
R2:	17	0.243		0.0500
R3:	17	0.042	•	0.0290

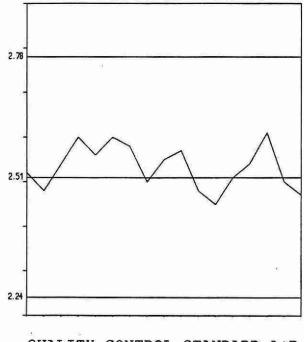
DUPLICATES:

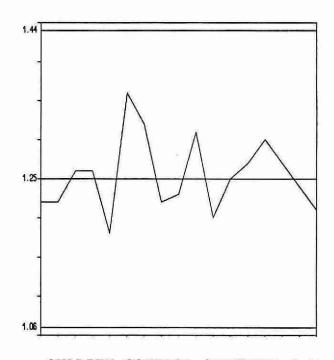
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
********	-				***************************************
10	0.00	-	0.50	0.0226	25.1
5	0.50		1.25	0.0630	6.0
23	1.25	· Es	2.50	0.1210	6.2
38	(Overall		0.0893	

	Number of Data	Data Mean	Standard(1) Deviation
Digested Blank	17	0.0006	0.0024

ALUMINUM, EXCHANGEABLE CATION (meq/100 g as Al)

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

*** ALUMINUM, REACTIVE SPECIES ***

IDENTIFICATION:

Laboratory

LIS Test Name Code Work Station Code Method Code

: Dorset : ALEXCV, ALNDCV : DOALSP

Method Introduced Units Unit Code Supervisor

: 24/10/85 : ug/L as Al : 063813 : A. Neary

Sample Type/Matrix

: 0928C2 : Streams, Lakes, and Soil Leachates

SAMPLING:

Quantity Required

: 30 mL

Container

: Plastic or glass

ANALYTICAL PROCEDURE:

The procedure is based on the formation of an aluminum catechol-violet complex at pH 6.2. Phenanthroline hydroxylamine HCl reagents are used to reduce interference by iron. An ion exchange column is used for separating organic and inorganic aluminum. Concentrations of aluminum are determined by comparison with a similarly prepared series of standards and reported as ug/L as CV reactive Al.

INSTRUMENTATION:

Automated auto-analyzer/sampler system with colourimeter and chart recorder.

REPORTING:

Maximum Significant Figures: 3

Current W value: 2,2

T value: 10,10

CALIBRATION:

BL plus 10 standards daily

CONTROLS:

Calibration

: LTBL plus 4 standards, e.g. QCA

Drift

: BL every 10 samples and BL plus check standard every 20 samples

ALUMINUM, REACTIVE SPECIES (ALEXCV)

QUALITY CONTROL DATA FROM 08/01/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 1000 ug/L as Al

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation

A :	52	750.0	751.1	1.1	5.87
B :	52	250.0	240.3	-9.7	5.95
A+B:	52	1000.0	991.4	-8.6	9.35
A-B:	52	500.0	510.9	10.9	7.24
C :	52	75.0	74.4	-0.6	2.91
D:	52	25.0	24.5	-0.5	2.28
C+D:	52	100.0	98.9	-1.1	3.97
C-D:	52	50.0	49.9	-0.1	3.40

s.d.(AB) S(between runs): 5.91

Sw(within run): 5.11 S/Sw: 1.15

s.d.(AB) S(between runs): 2.61

Sw(within run): 2.40 S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

963	4	1037	for	A+B
475	14	525	for	A-B
85		115	for	C+D
40		60	for	C-D

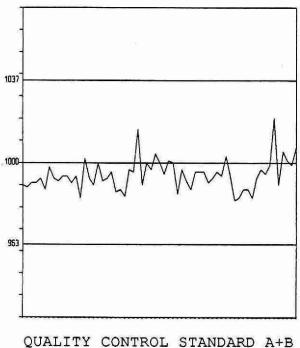
DUPLICATES:

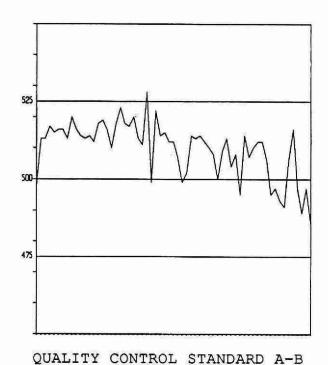
Number of	Sample		Mean(2)	Coefficient of var.(%)	
Data Pairs	Concn Span		s.d.		
60	0	5 0	50	3.90	22.7
36	50	5	100	10.71	13.7
40	100		400	9.34	6.1
0 136	400	- Overal	1000	N.A. 7.09	N.A.

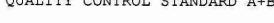
	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	52	0.327	1.216

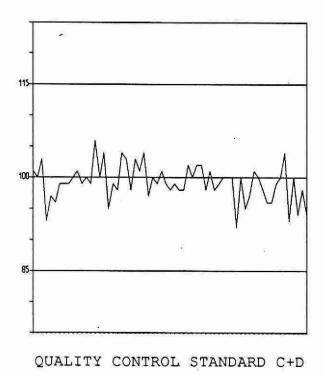
ALUMINUM, REACTIVE SPECIES (ug/L as Al)

QUALITY CONTROL DATA FROM 08/01/90 TO 21/09/90









QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

ALUMINUM, REACTIVE SPECIES (ALNDCV)

QUALITY CONTROL DATA FROM 08/01/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 1000 ug/L as Al

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
A :	63	750.0	752.1	2.1	4.36
B :	63	250.0	242.4	-7.6	6.67
A+B:	63	1000.0	994.6	-5.4	6.88
A-B:	63	500.0	509.7	9.7	8.92
C :	63	75.0	73.9	-1.1	1.68
D :	63	25.0	25.2	0.2	2.35
C+D:	63	100.0	99.2	-0.8	2.87
C-D:	63	50.0	48.7	-1.3	2.92

s.d.(AB) S(between runs): 5.63

Sw(within run): 6.31 S/Sw: 0.89

s.d.(AB) S(between runs): 2.05

Sw(within run): 2.07 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

963	1.	1037	for	A+B
475	-	525	for	A-B
85	-	115	for	C+D
40	-	60	for	C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
47	0	-1	50	4.36	17.3
39	50	= 1	100	6.25	7.7
49	100	- 5	250	9.93	7.2
32	250	1	500	15.17	5.6
7	500	100	1000	32.14	4.4
174		Overal	1	8.37	2

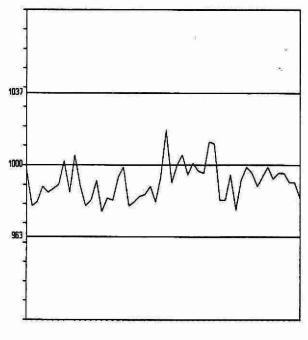
٠	Number of Data	Data Mean	Standard(1) Deviation

Long Term Blank	63	0.889	1.627

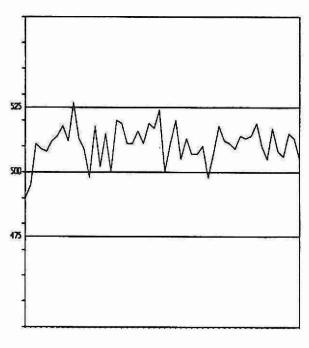
ALUMINUM, REACTIVE SPECIES (ug/L as AL)

(ALNDCV)

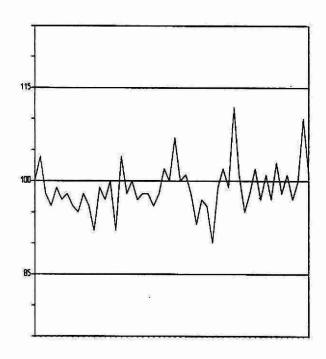
QUALITY CONTROL DATA FROM 08/01/90 TO 21/09/90



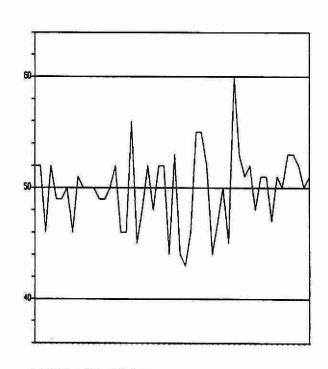
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** ALUMINUM, SODIUM PYROPHOSPHATE EXTRACTABLE ***

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: ALEPY : DOMETALX Units

: % by weight Al

Work Station Code Method Code

: DOMETAL : 703AA5 Unit Code

: 070813

Sample Type/Matrix

: Soil

Supervisor

: A. Neary

SAMPLING:

Quantity Required

: 0.5 g dry

Container

: Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2mm and a subsample ground to <500 um (35 mesh)

ANALYTICAL PROCEDURE:

A 0.300 g quantity of sample plus 30 mL of 0.1 M sodium pyrophosphate is agitated overnight in a centrifuge tube. Samples are centrifuged at 20,000 rpm for 15 minutes and the supernatant is analyzed by AAS at 309.3 nm with a NO_2 -acetylene flame.

Approximate absorbance: 0.3 at the full scale level

N.B. Iron and manganese may be determined on the same extract.

INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration:

Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of

full scale; 2 method blanks; round robin ECSS samples.

Drift:

BL plus 1 standard (100% F.S.) every 10 samples

NOTES:

Values for recoveries are unknown - average value used.

ALUMINUM, SODIUM PYROPHOSPHATE EXTRACTABLE

QUALITY CONTROL DATA FROM 26/03/90 TO 13/11/90

Lab: Dorset Soils

Analytical Range: - to 0.5 % by wt. Al

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	**********		**********		
A :	4	0.375	0.365	-0.010	0.0129
B :	4	0.125	0.122	-0.003	0.0095
A+B:	4	0.500	0.487	-0.013	0.0206
A-B:	4	0.250	0.242	-0.008	0.0096

s.d.(AB) S(between runs): 0.011

Sw(within run): 0.007 S/Sw: 1.7

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.46 - 0.54 for A+B 0.23 - 0.27 for A-B

RECOVERIES:

Number of Data		Av. Concn Measured	Standard(1) Deviation	
	**		***********	
R1:	4	9	0.287	0.017
R2:	4		0.420	0.022
R3:	4	₩.	0.282	0.017

DUPLICATES:

Number of Data Pairs	~ c	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
	****			*******	************
7	0.00		0.10	0.008	10.1
3	0.10	-	0.25	0.000	0.0
2	0.25	•	0.50	0.005	1.7
12	(Overall	Ļ	0.006	

	Number of Data	Data Mean	Standard(1) Deviation

Method Blank	4	0.000	0.000

*** ALUMINUM, SOLUBLE ***

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: ALECA

Units

: ug/g as Al (dried)

Work Station Code Method Code : DOSOLAL : 3144A5 Unit Code Supervisor : 073813 : A. Neary

Sample Type/Matrix

: Soil

SAMPLING:

Quantity Required

: 20 g (dry <2 mm)

Container

: Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

A 10 g sample plus 20 mL 0.01 M calcium chloride is agitated for 5 minutes, centrifuged and filtered. The filtration is analyzed for Al by AAS at 309.3 nm using an NO₂-acetylene flame. Approximate absorbance: 0.1 at the full scale level

INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.2

T value: 1.0

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration:

Three soil samples representing different soil types plus two solution controls at 10% and

30% of full scale and two method blanks.

Drift:

BL plus 1 standard (100%) every 10 samples

NOTES:

Values for recoveries are unknown - average value used.

ALUMINUM, SOLUBLE

QUALITY CONTROL DATA FROM 20/02/90 TO 27/11/90

Lab: Dorset Soils

Analytical Range: - to 40.0 ug/g as Al

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	13	30.0	29.97	-0.03	0.996
B :	13	10.0	9.45	-0.55	0.572
A+B:	13	40.0	39.43	-0.57	1.349
A-B:	13	20.0	20.52	0.52	0.905

s.d.(AB) S(between runs): 0.81

Sw(within run): 0.64 S/Sw: 1.27

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 35.2 44.8 for A+B 16.8 23.2 for A-B

RECOVERIES:

3	Number of Data	Av. Concn Measured	Standard(1) Deviation

R1:	13	3.37	0.491
R2:	- 13	12.27	1.035
R3:	13	31.88	4.263

DUPLICATES:

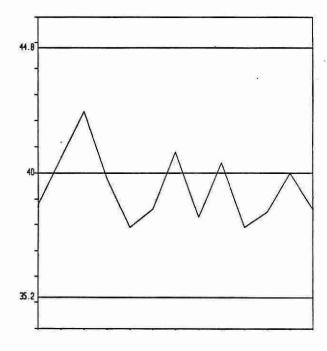
Number of Data Pairs				Mean(2) s.d.	Coefficient of var.(%)
*********					************
12	0.0	3 —	1.0	0.302	74.6
15	1.0		10.0	0.698	18.1
7	10.0	11.5	40.0	2.199	10.3
34		Overal	1	0.740	,,,,,,

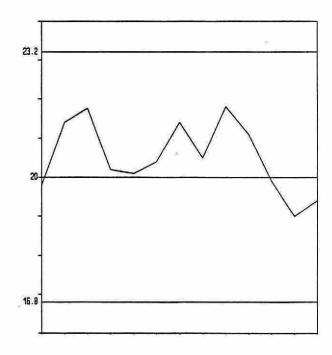
	Number of Data	Data Mean	Standard(1) Deviation

Method Blank	13	0.0008	0.003

ALUMINUM, SOLUBLE (ug/g as Al)

QUALITY CONTROL DATA FROM 20/02/90 TO 27/11/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

*** ALUMINUM, TOTAL ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 06/09/83

LIS Test Name Code Work Station Code : ALUT : DOAAS Units Unit Code : ug/L as Al : 063813

Method Code

: 005AF2

Supervisor

: A. Neary

Sample Type/Matrix

: Streams, Lakes, Precipitation, Biota and Groundwaters

SAMPLING:

Quantity Required

: 1 mL

Container

: 15 mL Polystyrene Tube, capped, acidified to 0.25% with HNO₃

ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 309.3 nm.

Approximate absorbance: .5 at the full scale level

INSTRUMENTATION:

Automated GFAAS/sampler system with microcomputer data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

CALIBRATION:

BL plus 5 standards daily

CONTROLS:

Calibration

: LTBL plus 4 standards, e.g. QCA

ALUMINUM, TOTAL

QUALITY CONTROL DATA FROM 18/01/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 200 ug/L as Al

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
			*********	**********	***********
A :	70	140.0	136.39	-3.61	7.16
B :	70	70.0	73.71	3.71	4.91
A+B:	70	210.0	210.10	0.10	8.40
A-B:	70	70.0	62.67	-7.33	8.96
C :	70	35.0	38.70	3.70	4.91
D:	70	7.0	7.73	0.73	3.76
C+D:	70	42.0	46.43	4.43	4.20
C-D:	70	28.0	30.97	2.97	3.66

s.d.(AB) S(between runs): 6.14

Sw(within run): 6.34 S/Sw: 0.97

s.d.(AB) S(between runs): 2.78

Sw(within run): 2.59 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

180	-	240	for	A+B
50		90	for	A-B
27		57	for	C+D
18	=	38	for	C-D

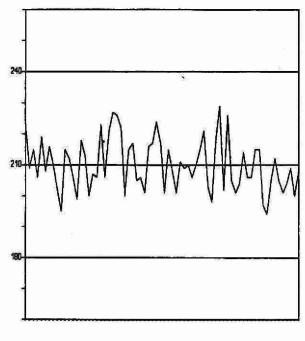
DUPLICATES:

Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)	
51	0.0	-	40.0	2.21	13.0	
30	40.0	14	100.0	3.77	6.5	
49	100.0	194	200.0	8.78	7.5	
130		Overal		4.60		

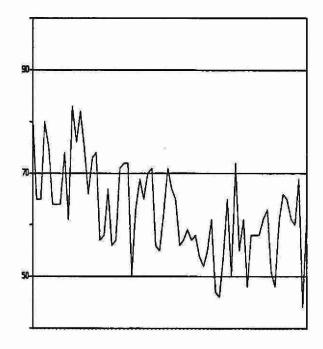
	Number	Data	Standard(1)
	of Data	Mean	Deviation
	~~ ~~ ~ ~ ~ ~ ~		
Long Term Blank	70	0	0

ALUMINUM, TOTAL (ug/L as A1)

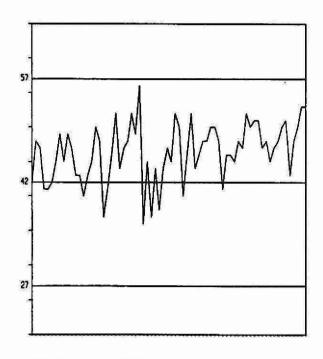
QUALITY CONTROL DATA FROM 18/01/90 TO 21/12/90



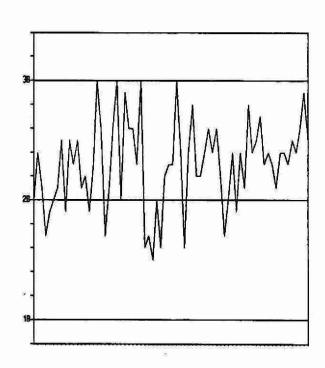
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** CADMIUM, TOTAL ***

IDENTIFICATION:

Laboratory LIS Test Name Code

: Dorset : CDUT

Method Introduced Units

: 26/11/84 : ug/L as Cd : 063848

Work Station Code Method Code

: DOAAS : 005AF2

Unit Code Supervisor

: A. Neary

Sample Type/Matrix

: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required Container

: 1 mL

: 500 mL acid washed Teflon container, bagged in a clean room

ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 228.8 nm.

Approximate absorbance: .400 at the full scale level

INSTRUMENTATION:

Automated GFAAS/sampler system with microcomputer data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

CALIBRATION:

BL plus 4 standards daily

CONTROLS:

Calibration: LTBL plus 4 standards, e.g. OCA

CADMIUM, TOTAL

QUALITY CONTROL DATA FROM 09/01/90 TO 19/12/90

Lab: Dorset

Analytical Range: - to 2.0 ug/L as Cd

CALIBRATION CONTROL:

19	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	17	0.160	0.156	-0.004	0.0336
B :	17	0.060	0.051	-0.009	0.0110
A+B:	17	0.220	0.207	-0.013	0.0428
A-B:	17	0.100	0.105	0.005	0.0258

s.d.(AB) S(between runs): 0.025

Sw(within run): 0.018 S/Sw: 1.37

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.09 - 0.35 for A+B -0.01 - 0.21 for A-B

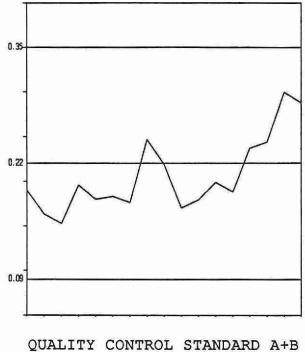
DUPLICATES:

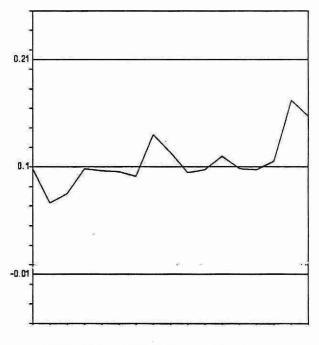
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*******				***********	************
14	0.000	i a	0.025	0.010	49.2
19	0.025		0.100	0.014	24.4
14	0.100	*	0.500	0.024	12.8
0	0.500	•	2.000	N.A.	N.A.
47	C	vera		0.016	

	Number of Data	Data Mean	Standard(1) Deviation
	/ 		
Long Term Blank	17	0.006	0.0074

CADMIUM, TOTAL (ug/L as Cd)

QUALITY CONTROL DATA FROM 09/01/90 TO 19/12/90





QUALITY CONTROL STANDARD A-B

*** CALCIUM ***

IDENTIFICATION:

Laboratory

Lis Test Name Code

: Atomic Absorption : CAUR

Method Introduced Units

: 18/05/79 : mg/L as Ca

Work Station Code Method Code: : PRAA400 : 002CA1 Unit Code Supervisor : 064820 : M. Young

Sample Type/Matrix

: Precipitation, Throughfall

SAMPLING:

Quantity Required Container

i : 5 mL : Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.2 at the full scale level.

INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g., QCA

Drift

: BL, reslope standard every 10 samples.

CALCIUM

QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90

Lab: Atomic Absorption

Analytical Range: - to 2.0 mg/L as Ca

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	78	1.20	1.198	-0.002	0.0102
B :	78	0.20	0.201	0.001	0.0042
A+B:	78	1.40	1.399	-0.001	0.0121
A-B :	78	1.00	0.997	-0.003	0.0098

s.d.(AB) S(between runs): 0.0078

Sw(within run): 0.0070 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.31 - 1.49 for A+B 0.94 - 1.06 for A-B

DUPLICATES:

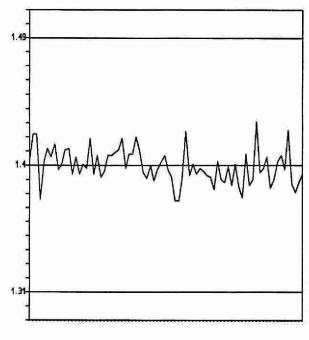
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
123	0.00		0.20	0.004	5.9
66	0.20		0.50	0.007	2.4
15	0.50	-	1.00	0.010	1.3
10	1.00		2.00	0.012	0.9
214	C	Overall		0.006	

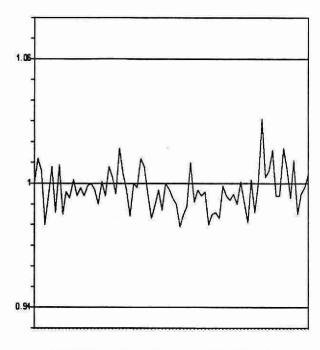
	Number	Data	Standard(1)
	of Data	Mean	Deviation

Long Term Blank	78	-0.0010	0.0051

CALCIUM (mg/L as Ca)

QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

*** CALCIUM ***

IDENTIFICATION:

Laboratory

: Atomic Absorption

Method Introduced

: 20/07/88

LIS Test Name Code Work Station Code

: CAUR : PRAAS Units Unit Code : mg/L as Ca : 064820

Method Code Sample Type/Matrix : 002CA1 : Rivers, Lakes Supervisor

: M. Young

SAMPLING:

: 5 mL

Quantity Required Container : Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.2 at the full scale level.

INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration

: LTBL plus 3 standards, e.g., QCA

Drift

: BL, reslope standard every 10 samples.

CALCIUM

QUALITY CONTROL DATA FROM 04/01/90 TO 21/12/90

Lab: Atomic Absorption

Analytical Range: - to 8.0 mg/L as Ca

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	100	6.4	6.399	-0.001	0.0376
В:	100	1.6	1.613	0.013	0.0155
A+B:	100	8.0	8.012	0.012	0.0464
A-B:	100	4.8	4.786	-0.014	0.0339
C :	100	1.6	1.613	0.013	0.0066
D:	100	0.4	0.408	0.008	0.0464
C+D:	100	2.0	2.021	0.021	0.0133
C-D:	100	1.2	1.205	0.005	0.0065

s.d.(AB) S(between runs): 0.029 Sw(within run): 0.024 S/Sw: 1.2

s.d.(CD) S(between runs): 0.012 Sw(within run): 0.009 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

7.64 - 8.36 for A+B 4.65 - 4.95 for A-B 1.64 - 2.36 for C+D 1.05 - 1.35 for C-D

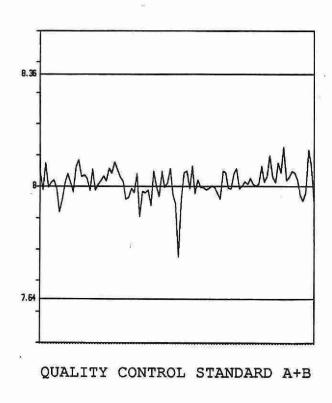
DUPLICATES:

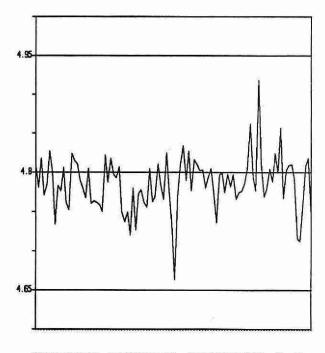
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	

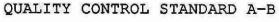
34	0.00	=	1.60	0.0178	2.3
124	1.60		3.00	0.0289	1.3
115	3.00	18	8.00	0.0576	1.5
273	(Overal		0.0393	o screp

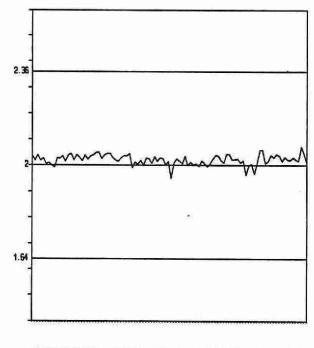
	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	100	-0.0037	0.0066

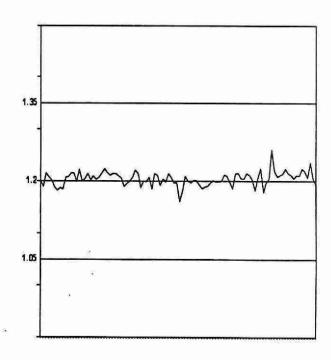
QUALITY CONTROL DATA FROM 04/01/90 TO 21/12/90











QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

*** CALCIUM ***

IDENTIFICATION:

Laboratory

: Atomic Absorption

Method Introduced

: 01/04/74

Lis Test Name Code Work Station Code : CAUR : RMAAS Units Unit Code : mg/L as Ca : 064820

Method Code Sample Type/Matrix : 0901A1 : Rivers, Lakes, Soil Extracts Supervisor

: M. Young

SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.14 at the full scale level.

INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.1

T value: 0.5

CALIBRATION:

BL plus 11 standards

CONTROLS:

Calibration

: LTBL plus 3 standards, e.g., QCA

Drift

: BL every 10 samples; 2 standards every 20 samples.

CALCIUM

QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90

Lab: Atomic Absorption

Analytical Range: - to 40.00 mg/L as Ca

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation

A :	97	32.0	31.66	-0.34	0.3527
B :	97	8.0	7.96	-0.04	0.1224
A+B:	97	40.0	39.62	-0.38	0.4065
A-B:	97	24.0	23.70	-0.30	0.3369
C :	97	8.0	7.96	-0.04	0.1224
D:	97	2.0	1.99	-0.01	0.0506
C+D:	97	10.0	9.95	-0.05	0.1471
C-D:	97	6.0	5.98	-0.02	0.1160

s.d.(AB) S(between runs): 0.26 Sw(within run): 0.24 S/Sw: 1.1

s.d.(CD) S(between runs): 0.09 Sw(within run): 0.08 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

38.35 - 41.65 for A+B 22.90 - 25.10 for A-B 9.25 - 10.75 for C+D 5.50 - 6.50 for C-D

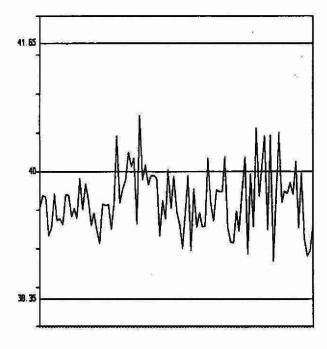
DUPLICATES:

Number of Data Pairs	C	Samp oncn		Mean(2) s.d.	Coefficient of var.(%)
**********	*****				*****
40	0.00	13	2.00	0.0350	3.2
63	2.00	-	5.00	0.0606	2.0
46	5.00	-	20.00	0.1242	1.2
43	20.00	-	40.00	0.2913	1.2
192	(Overal		0.0976	

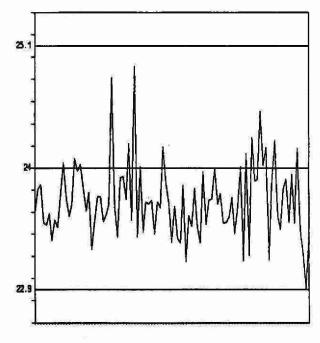
	Number of Data	Data Mean	Standard(1) Deviation
	***********	00 0000 0000 0000	
Long Term Blank	95	-0.0028	0.0293

CALCIUM (mg/L as Ca)

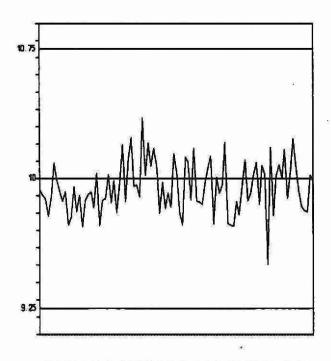
QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90



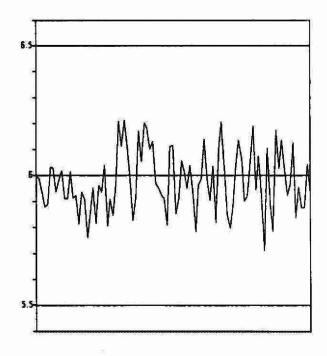
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** CALCIUM ***

IDENTIFICATION:

Laboratory

: Atomic Absorption

Method Introduced

: 08/04/86

Lis Test Name Code Work Station Code : CAUR : WAAS Units Unit Code

: mg/L as Ca : 064820

Method Code Sample Type/Matrix : 002CA1

Supervisor

: M. Young

oumpre rypermune

: Domestic Waters, Leachates, Effluents, Sewage, Industrial Wastes

SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm using an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.17 at the full scale level.

INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

BL plus 11 standards

CONTROLS:

Calibration

: LTBL plus 3 standards e.g. QCA

Drift

: BL every 10 samples; 2 standards every 20 samples

CALCIUM

QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90

Lab: Atomic Absorption

Analytical Range: - to 200.0 mg/L as Ca

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*********	**********		*********	*********
A :	138	160.0	159.8	0.2	2.820
B :	138	40.0	40.03	0.03	1.022
A+B:	138	200.0	199.8	0.2	3.209
A-B:	138	120.0	119.8	0.2	2.774
C :	138	40.0	40.03	0.03	1.022
D:	138	10.0	9.94	0.06	0.427
C+D:	138	50.0	49.97	0.03	1.175
C-D:	138	30.0	30.08	0.08	1.037

s.d.(AB) S(between runs): 2.12 Sw(within

Sw(within run): 1.96 S/Sw: 1.1

s.d.(CD) S(between runs): 0.78

Sw(within run): 0.73 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

190 - 210 for A+B 113 - 127 for A-B 44.5 - 54.5 for C+D 27.0 - 33.0 for C-D

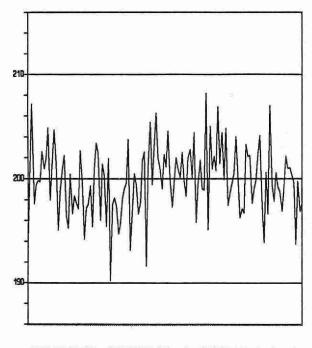
DUPLICATES:

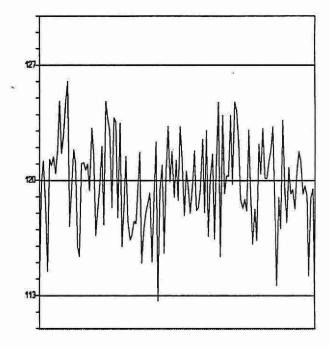
Number of Data Pairs	Sample Concn Span					Coefficient of var.(%)
	-	*****				
41	0.00	-	10.00	0.2456	13.7	
22	10.00	•	20.00	0.4546	2.7	
83	20.00	-	50.00	0.9648	2.6	
119	50.00	-	100.00	1.6227	2.2	
91	100.00	-	200.00	2.7094	2.4	
356		Overa		1.4125	No salah	

	Number of Data	Data Mean	Standard(1) Deviation

Long Term Blank	129	-0.1669	0.5837

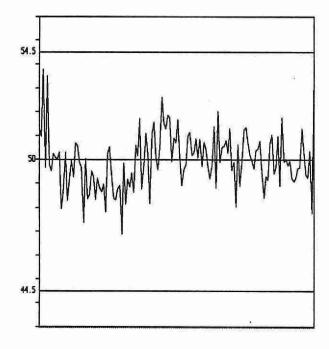
QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B



30

QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

*** CALCIUM, EXCHANGEABLE CATION ***

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: CAESC

Units Unit Code : meq/100 g : 355000

Work Station Code Method Code : DOCATION : 306AA1

Supervisor

: A. Neary

Sample Type/Matrix

: Soil

SAMPLING:

Quantity Required

: 6 g dry

Container

: Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for Ca by AAS at 422.7 nm with an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale level.

Aluminum, magnesium, and potassium are determined on the same extract.

INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration:

Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of full

scale; 2 method blanks; round robin ECSS samples (run occasionally).

Drift:

BL plus 1 standard (100% F.S.) every 10 samples

NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown - average value used.

CALCIUM, EXCHANGEABLE CATION

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90

Lab: Dorset Soils

Analytical Range: - to 5.0 meq/100 g

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	****		************		***************************************
A :	17	3.75	3.780	0.030	0.0665
B :	17	1.25	1.211	-0.039	0.0438
A+B:	17	5.00	4.992	-0.008	0.0935
A-B :	17	2.50	2.569	0.069	0.0630

s.d.(AB) S(between runs): 0.056 Sw(within run): 0.044 S/Sw: 1.26

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.63 - 5.37 for A+B 2.25 - 2.75 for A-B

RECOVERIES:

Number of Data		Av. Concn Measured	Standard(1) Deviation	
R1:	17	2.998	0.170	
R2:	17	1.709	0.151	
R3:	17	0.534	0.077	

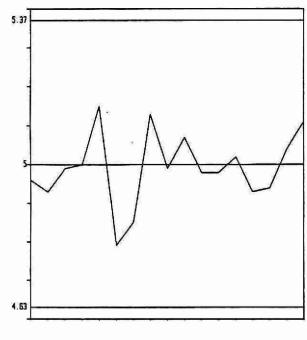
DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
5	0.0	-	1.0	0.058	6.2
20	1.0		2.5	0.138	12.6
25	2.5	-	5.0	0.188	5.5
50	O	veral	1	0.159	100 (100)

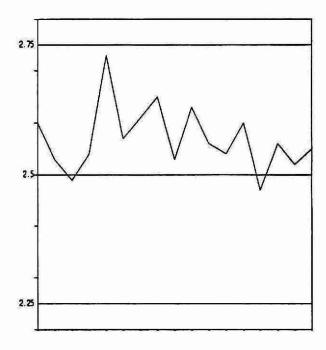
	Number of Data	Data Mean	Standard(1) Deviation
	***********	***********	
Digested Blank	17	0.0006	0.002

CALCIUM, EXCHANGEABLE CATION (meq/100 g as Ca)

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

*** CARBON, DISSOLVED INORGANIC ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 03/06/80

LIS Test Name Code Work Station Code : DIC : DODIC Units Unit Code : mg/L as C : 064806

Method Code

: 1127C2

Supervisor

: A. Neary

Sample Type/Matrix

: Streams, Lakes, and Soil Leachates

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO₂-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

CALIBRATION:

BL plus 9 standards daily

CONTROLS:

Calibration:

LTB plus 4 standards, e.g. QCA, QCB, QCC, QCD

Drift:

BL every 10 samples; BL plus 1 check standard every 20 samples

NOTES:

As concentrations of calibration control solutions slowly change with time at these low concentrations, calibration control ranges are based on long term measured averages rather than expected concentrations. This method was changed to incorporate DCI in Jan. 1989.

CARBON, DISSOLVED INORGANIC

QUALITY CONTROL DATA FROM 05/01/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 10.00 mg/L as C

CALIBRATION CONTROL:

8	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	128	7.50	7.44	-0.06	0.111
B :	128	2.25	2.17	-0.08	0.059
A+B:	128	9.75	9.61	-0.14	0.136
A-B:	128	5.25	5.26	0.01	0.114
C :	128	1.50	1.44	-0.06	0.052
D:	128	0.50	0.49	-0.01	0.043
C+D:	128	2.00	1.94	-0.06	0.083
C-D:	128	1.00	0.96	-0.04	0.048

s.d.(AB) S(between runs): 0.09 Sw(within run): 0.08 S/Sw: 1.1

s.d.(AB) S(between runs): 0.05 Sw(within run): 0.03 S/Sw: 1.4

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.15 - 10.35 for A+B 4.85 - 5.65 for A-B 1.70 - 2.30 for C+D 0.80 - 1.20 for C-D

DUPLICATES:

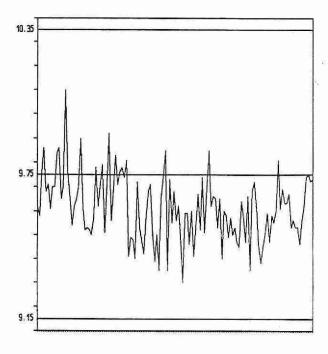
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
				********	*************
51	0.00		0.50	0.016	6.3
77	0.50	Ħ	1.00	0.027	3.1
116	1.00	+	2.00	0.038	2.3
119	2.00	#	5.00	0.091	3.0
20	5.00	-	10.00	0.175	2.3
383	(Overall	C	0.052	

	Number of Data	Data Mean	Standard(1) Deviation

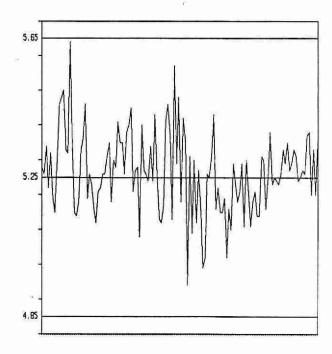
Long Term Blank	128	0.210	0.0448

CARBON, DISSOLVED INORGANIC (mg/L as C)

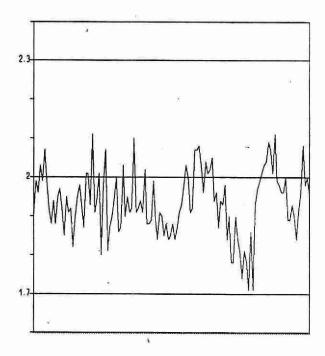
QUALITY CONTROL DATA FROM 05/01/90 TO 21/12/90



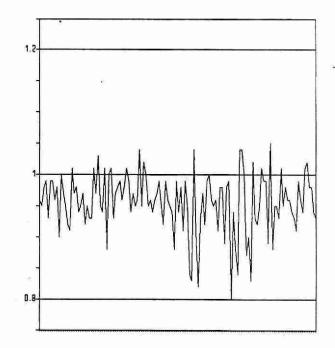
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** CARBON, DISSOLVED INORGANIC ***

IDENTIFICATION:

Laboratory

Lis Test Name Code

Work Station Code Method Code : Colourimetry : DIC

Method Introduced Units : 01/04/78 : mg/L as C

: ROM : 102AC2

Unit Code Supervisor : 064806 : M. Rawlings

Sample Type/Matrix

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies,

Leachates, Sewages, Industrial Wastes

SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved organic carbon, and reactive silicates are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO₂-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

CARBON, DISSOLVED INORGANIC

QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 40.0 mg/L as C

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
A :	208	32	32.04	0.04	0.552
B :	208	8	7.83	-0.17	0.241
A+B:	208	40	39.85	-0.15	0.700
A-B:	208	24	24.20	0.20	0.484
C :	208	8	7.83	-0.17	0.241
D:	208	2	1.86	-0.14	0.127
C+D:	208	10	9.69	-0.31	0.320
C-D:	208	6	5.96	-0.04	0.215

s.d.(AB) S(between runs): 0.43 Sw(within run): 0.34 S/Sw: 1.2

s.d.(CD) S(between runs): 0.19 Sw(within run): 0.15 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

37.80 - 42.20 for A+B 22.50 - 25.50 for A-B 8.90 - 11.10 for C+D 5.30 - 6.70 for C-D

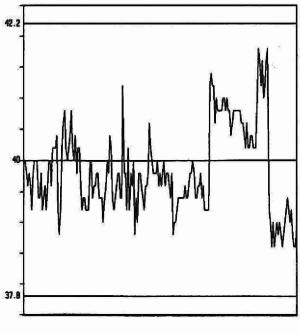
DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
~~~~~~				*******	*******
79	0.0	-	1.0	0.156	75.3
32	1.0	-	2.0	0.294	20.8
141	2.0	•	20.0	0.305	6.0
158	20.0	I.	40.0	0.351	1.8
410		Overall		0.288	B. (1971)

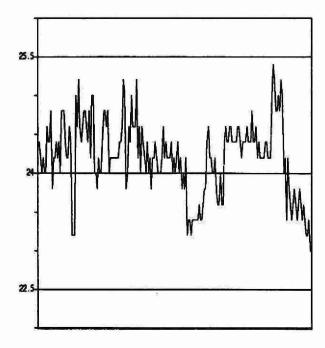
	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	208	-0.059	0.140

# CARBON, DISSOLVED INORGANIC (mg/L as C)

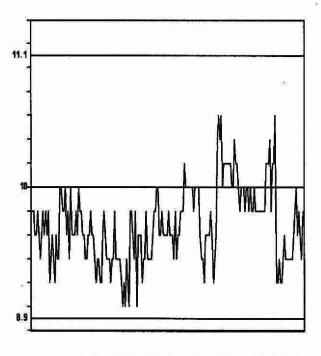
QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90



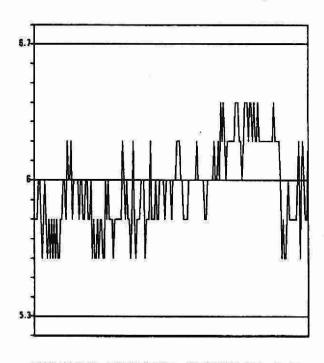
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# *** CARBON, DISSOLVED ORGANIC ***

#### **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code

Method Code

: Colourimetry : DOC

Method Introduced

Units
Unit Code
Supervisor

: mg/L as C ode : 064806 isor : M. Rawlings

: 01/04/78

Sample Type/Matrix

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies,

Leachates, Sewages, Industrial Wastes

#### SAMPLING:

Quantity Required

: 10 mL

: ROM

: 102AC2

Container

: Glass or plastic

## ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas (500 mL/min) to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light (UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

Dissolved inorganic carbon, and reactive silicates are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO₂-free) supplies with flow controls, dialysis unit, UV digestor. Colourimetric measurement is through a 5.0 cm. light path at 550 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.1

T value: 0.5

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; 2 standards every 20 samples

#### NOTES:

1990 graphs of the quality control data revealed an out of control value in A-B, and corresponding C+D quality control variates. This was shown to be due to a deteriorated control standard B and the affected run was reported normally after confirmation.

# CARBON, DISSOLVED ORGANIC

# QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 20.0 mg/L as C

## CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
			********		
<b>A</b> :	210	16.0	16.01	0.01	0.12
B :	210	4.0	3.95	-0.05	0.09
A+B:	210	20.0	19.95	-0.05	0.16
A-B:	210	12.0	12.06	0.06	0.14
C :	210	4.0	3.95	-0.05	0.09
D:	210	1.0	0.99	-0.01	0.08
C+D:	210	5.0	4.94	-0.06	0.15
C-D:	210	3.0	2.96	-0.04	0.07

s.d.(AB) S(between runs): 0.11

Sw(within run): 0.10 S/Sw: 1.1

s.d.(CD) S(between runs): 0.08

Sw(within run): 0.05 S/Sw: 1.6

On any given day the calibration is accepted if the values obtained lie within the ranges:

19.30		20.70	for	A+B
11.50	1000	12.50	for	A-B
4.60	-	5.40	for	C+D
2.76		3 24	for	C-D

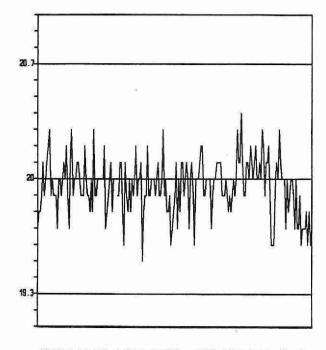
## **DUPLICATES:**

Number of Data Pairs	C	Samp oncn		Mean(2) s.d.	Coefficient of var.(%)
	****				*************
182	0.00	₹ <b>*</b>	2.00	0.098	24.3
196	2.00		4.00	0.107	11.4
185	4.00	2 <b></b>	10.00	0.163	3.8
29	10.00	a <del>st</del>	20.00	0.284	2.7
592	(	Overal	1	0.129	

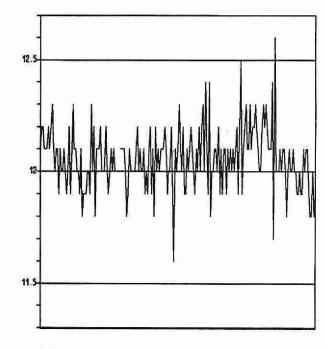
	Number of Data	Data Mean	Standard(1) Deviation
			***********
Long Term Blank	211	0.014	0.092

# CARBON, DISSOLVED ORGANIC (mg/L as C)

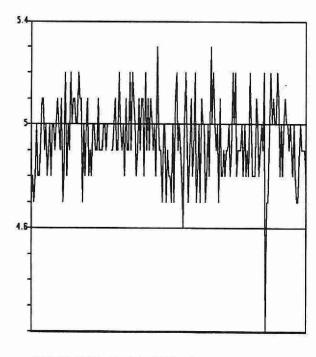
QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90



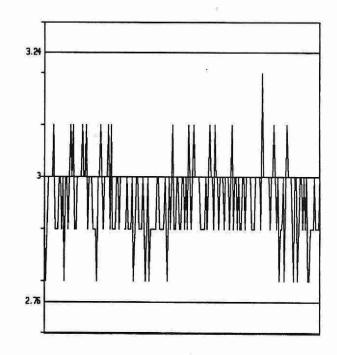
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## *** CARBON, TOTAL ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

LIS Test Name Code Work Station Code : ORGC : DOOXMAT : CALCO1

Method Code Sample Type/Matrix Method Introduced

: 01/10/80

Units

Unit Code

: % organic carbon : 500806

: 500

Supervisor

: A. Neary

#### SAMPLING:

Quantity Required

: 0.1 to 0.5 g dry

Container

: Glass

: Soil

#### SAMPLE PREPARATION:

Samples are air dried and a <2mm subsample ground to <500um.

#### **ANALYTICAL PROCEDURE:**

Total carbon is determined by a UIC/Coulometrics combustion furnace with electrometric titration. The percentage by weight of organic carbon in a soil sample is reported and is calculated as the difference between total carbon and inorganic carbon. Inorganic carbon (carbonate C) is determined coulometrically after reaction of the sample in HCl.

#### INSTRUMENTATION:

-UIC/Coulometrics combustion furnace with coulometric titration of carbon

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

#### CONTROLS:

CaCO3, plus 2 representative soil samples, 3 duplicates

# CARBON, TOTAL

# QUALITY CONTROL DATA FROM 08/03/90 TO 07/11/90

Lab: Dorset Soils

Analytical Range: - to 40.0 % organic carbon

# **RECOVERIES:**

	Number	Av. Concn	Standard(1)
	of Data	Measured	Deviation
R1:	15	11.97	0.052
R2:	16	3.74	0.016
R3:	16	0.38	0.008

# **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
28	0.0	1000	5.0	0.156	5.4
14	5.0	-	10.0	0.488	6.6
4	10.0	-	40.0	1.473	5.9
46	(	Overa	11	0.471	

	Number of Data	Data Mean	Standard(1) Deviation
Filtered Blank	19	0.017	0.005

## *** CARBON, TOTAL CARBONATE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code Work Station Code

: TIC : DOTIC Units Unit Code : % dry weight as C

Method Code

: 002AB1

: 070806 Supervisor : A. Neary

Sample Type/Matrix

: Soil

#### SAMPLING:

Quantity Required

: 2 g dry

Container

: Glass

#### SAMPLE PREPARATION:

Samples are air dried and ground to <500 um.

#### ANALYTICAL PROCEDURE:

Inorganic carbon is determined by measuring the CO₂ evolved by the reaction of carbonate with hydrochloric acid in a closed system (constant temperature and pressure). The CO₂ is swept by purified air through a KI scrubber into the cathode compartment of a coulometer in which the CO₂ is absorbed by the cathode solution. It is measured by automated coulometric titration to a colourimetric endpoint.

#### **INSTRUMENTATION:**

- -Coulometrics 5010 CO2 coulometer
- -Carbonate impinger train Coulometrics
- -Balance, accurate to 0.0001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

#### CONTROLS:

Calcium Carbonate 12%

Barium Carbonate (6.1%)

Two soil samples representing different soil types and inorganic carbon concentrations.

## NOTES:

Inorganic carbon is not analyzed for samples in which pH  $(0.01M \text{ CaCl}_2) < 5.0$ .

## CARBON, TOTAL CARBONATE

## QUALITY CONTROL DATA FROM 27/02/90 TO 07/11/90

Lab: Dorset Soils

Analytical Range: - to 2.0 % as C

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	10	12.00	12.014	0.014	0.0574
B :	10	6.08	6.066	-0.014	0.0510
A+B:	10	18.08	18.080	0.000	0.0754
A-B:	10	5.92	5.948	0.028	0.0781

s.d.(AB) S(between runs): 0.054 Sw(within run): 0.055 S/Sw: 0.98

On any given day the calibration is accepted if the values obtained lie within the ranges:

17.77 5.68 18.39 6.15 for A+B A-B for

## RECOVERIES:

	Number of Data	Av. Concn Measured	Standard(1) Deviation
		******	
R1:	10	3.609	0.04458
R2:	9	0.010	0.00475

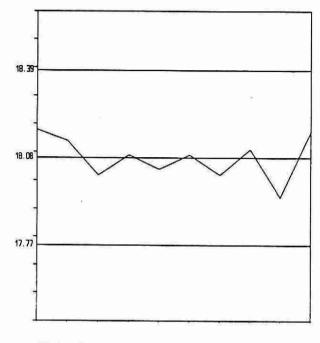
## **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
**********					
80	0.00	4	0.01	0.0014	24.7
18	0.01	<b>2</b>	1.00	0.0045	13.1
0	1.00	¥	2.00	N.A.	N.A
26	9	Overall		0.004	

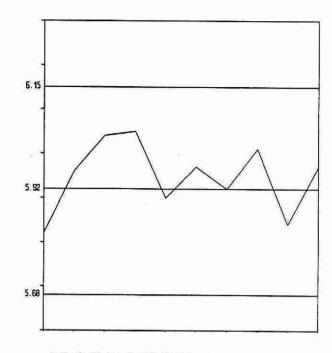
	Number of Data	Data Mean	Standard(1) Deviation
Digested Blank	10	0.0046	0.0021

# CARBON, TOTAL CARBONATE (% wt. as C)

QUALITY CONTROL DATA FROM 27/02/90 TO 07/11/90







QUALITY CONTROL STANDARD A-B

## *** CARBON, TOTAL ORGANIC ***

#### **IDENTIFICATION:**

Laboratory

: MISA

Method Introduced

: 01/05/89

LIS Test Name Code Work Station Code : TOC : WAC Units Unit Code : mg/L as C : 064000

Method Code

: SUM001

Supervisor

: P. Campbell

Sample Type/Matrix

: Industrial Effluents*, Sewage* (*Prior authorization required for this test)

#### SAMPLING:

Quantity Required

: 500 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

If particles in the sample are greater than about 2 mm diameter the sample is homogenized. An aliquot up to 100mL is acidified with sulphuric acid to pH 2.0, then bubbled with nitrogen gas for 10 minutes to remove inorganic carbon. The aliquot is then filtered through a 47 mm diameter glass fibre filter (particle size retention nominally 1.5 um). The filtrate is analyzed in an automated system, using ultra-violet/persulphate digestion with infra-red detection of carbon dioxide, to obtain the manually-acidified dissolved organic carbon (ADOC) result. The filter is dried at 103°C overnight and ignited at 1370°C in a Leco carbon analyzer to obtain the non-dissolved organic carbon (NDOC) result. The sum of ADOC and NDOC provides the TOC result.

#### INSTRUMENTATION:

Astro 2001 carbon analyzer with autosampler Leco CR12 carbon analyzer

## REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

#### CALIBRATION:

A solution of potassium biphthalate is used to calibrate the ADOC analyzer. Powdered potassium biphthalate standards are used to calibrate the NDOC analyzer.

#### CONTROLS:

Calibration:

ADOC: 3 QC solutions used until 30/04/90

ADOC: 4 QC solutions used beginning 01/05/90

NDOC: 2 powdered standards

Blanks:

ADOC: Untreated acidified distilled deionized water (LTB) and method blank

NDOC: Unused filters and method blank filters are used

Drift:

Standard every 10 crucibles in Leco or every 10 tubes in auto-sampler

Matrix: Répeat sample diluted 50% further at least every 10 samples

Spiked sample at least every 10 samples

Precision:

Duplicate sample at least every 10 samples

# *** CARBON, TOTAL ORGANIC cont'd

## **MODIFICATIONS:**

01/05/90 Another ADOC analytical range was added ( - to 10 mg/L as C) and 2 QC solutions were provided for each range.

## NOTES:

Extra QC checks were added for the MISA program.
Calibration control graphs are provided for the following:
01/01/90 to 30/04/90 for ADOC
01/05/90 to 31/12/90 for ADOC for both ranges
01/01/90 to 31/12/90 for NDOC
Variability of Blanks for ADOC is underestimated since instrument reports negative results as 0.00

## CARBON, TOTAL ORGANIC

# QUALITY CONTROL DATA FROM 01/01/90 TO 30/04/90

Lab: MISA

Analytical Range: - to 50 mg/L as C

## CALIBRATION CONTROL: ADOC

ADOC	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	****				************
A:	15	40.0	40.29	0.29	0.5365
B:	15	5.0	5.14	0.14	0.3673
A+B:	15	45.0	45.43	0.43	0.7353
A-B:	15	35.0	35.15	0.15	0.5522
C:	15	5.0	5.14	0.14	0.3673
D:	15	1.0	1.14	0.14	0.2478
C+D:	15	6.0	6.29	0.29	0.5552
C-D:	15	4.0	3.999	0.001	0.2905

# **ADOC**

s.d.(AB) S(between runs): 0.46 s.d.(CD) S(between runs): 0.20

Sw(within run): 0.39 Sw(within run): 0.31

S/Sw: 1.11

S/Sw: 1.52

On any given day the calibration is accepted if the values obtained lie within the ranges:

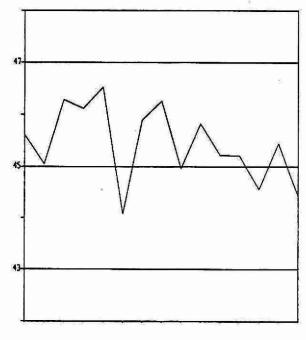
## ADOC

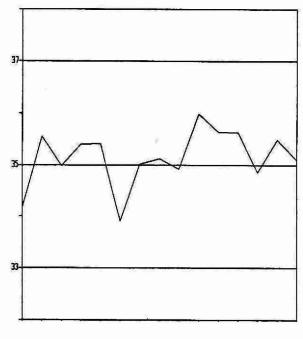
43.0	-	47.0	for	A+B
33.0	-	37.0	for	A-B
4.0		8.0	for	C+D
3.0	(-1	5.0	for	C-D

i.		Number of Data	Data Mean	Standard(1) Deviation
			-	
ADOC: LTB		15	0.2067	0.2428

# CARBON, TOTAL ORGANIC (mg/L as C)

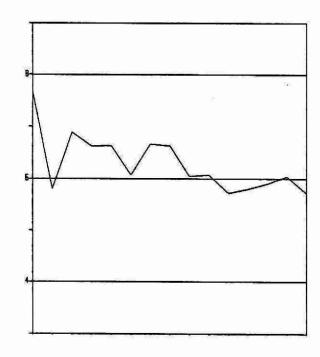
QUALITY CONTROL DATA FROM 01/01/90 TO 30/04/90

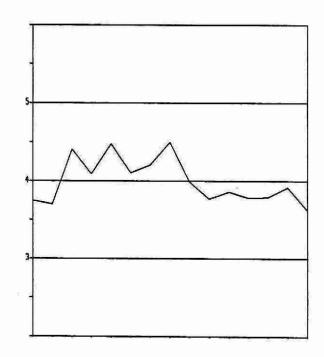




QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B





QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

_____CONTROL LIMIT

# CARBON, TOTAL ORGANIC

# QUALITY CONTROL DATA FROM 01/05/90 TO 31/12/90

Lab: MISA

Analytical Range: - to 50 mg/L as C

## CALIBRATION CONTROL: ADOC

ADOC	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		**********		~~~~~~~	*************
A:	45	40.0	39.74	0.26	0.8357
B:	45	10.0	9.96	0.04	0.3700
A+B:	45	50.0	49.70	0.30	0.8811
A-B:	45	30.0	29.78	0.22	0.9457

ADOC

s.d.(AB) S(between runs): 0.64

Sw(within run): 0.67

S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within the ranges:

**ADOC** 

47.4

for A+B

27.9

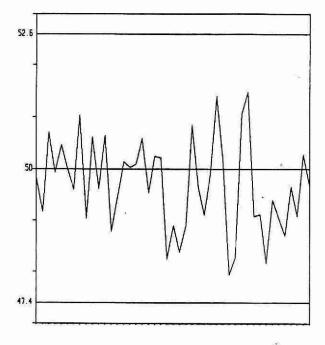
52.6 32.1

A-B

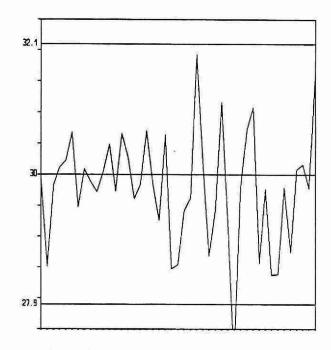
		Number of Data	Data Mean	Standard(1) Deviation
			· · · · · · · · · · · · · · · · · · ·	***********
ADOC:	MTD BLK LTB	27 42	0.1855 0.1307	0.3865 0.2963

# CARBON, TOTAL ORGANIC (mg/L as C)

QUALITY CONTROL DATA FROM 01/05/90 TO 31/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

## CARBON, TOTAL ORGANIC

## QUALITY CONTROL DATA FROM 01/05/90 TO 31/12/90

Lab: MISA

Analytical Range: - to 10 mg/L as C

# CALIBRATION CONTROL: ADOC

ADOC	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	********	**********			
<b>c</b> :	58	8.0	7.85	-0.15	0.2231
D:	58	2.0	2.18	0.18	0.1284
C+D:	58	10.0	10.02	0.02	0.2991
C-D:	58	6.0	5.67	-0.03	0.2072

**ADOC** 

s.d.(AB) S(between runs): 0.18

Sw(within run): 0.15

S/Sw: 1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

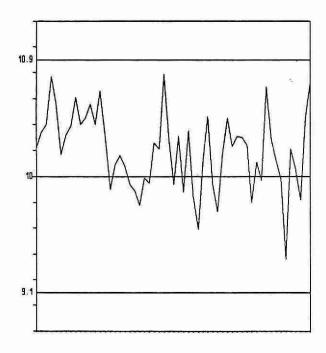
**ADOC** 

9.1 - 10.9 for C+D 5.4 - 6.6 for C-D

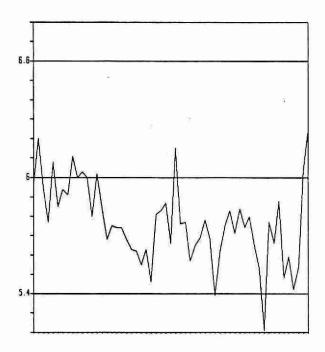
	Number	Data	Standard(1)
	of Data	Mean	Deviation
ADOC: LTB	6	0.0001	0.0006

# CARBON, TOTAL ORGANIC (mg/L as C)

QUALITY CONTROL DATA FROM 01/05/90 TO 31/12/90



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# CARBON, TOTAL ORGANIC

# QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

## CALIBRATION CONTROL: NDOC

NDO	C	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		******		***********		
14	A:	46	32.0	32.63	0.63	1.2932
	B:	46	12.0	12.13	0.13	0.7136
	A+B:	46	44.0	44.76	0.76	1.5212
	A-B:	46	20.0	20.49	0.49	1.4316
			*			

#### NDOC

s.d.(AB) S(between runs): 1.04	Sw(within run): 1.01				S/Sw: 1.03
NDOC					
	40	-	48	for	A+B
	17	74	23	for	A-B

# OTHER CHECKS:

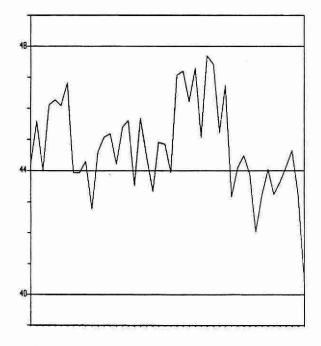
	Number of Data	Data Mean	Standard(1) Deviation
			*********
NDOC: MTD BLK NDOC: BLK	23 23	0.0068 0.0435	0.0201 0.0826

## **DUPLICATES:**

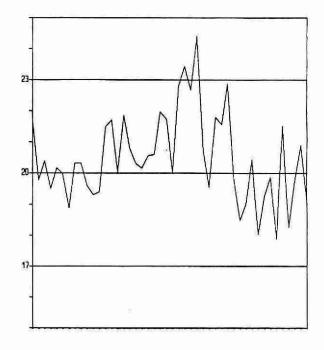
TOC	Number of Data Pairs		Sam Concn		Mean(2) s.d.	Coefficient of var.(%)
	14	0.0	-	1.0	0.1037	42.4
	91	1.0	-	5.0	0.2935	12.2
	75	5.0	4/	10.0	0.6920	14.1
	80	10.0	4	50.0	1.0014	7.1
	5	50.0	-	100.0	8.2313	9.8
	265		Overa	<u>l</u>	0.6114	7.55

# CARBON, TOTAL ORGANIC (mg/L as C)

# QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

#### *** CHLORIDE ***

## **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code

Method Code Sample Type/Matrix

: Colourimetry : CLIDUR

: COCL

Method Introduced Units

Unit Code

: 01/05/75 : mg/L as Cl : 064960 : M. Rawlings

: 004BC2 -Supervisor : Rivers (non-APIOS), Lakes (non-APIOS), Soil Extracts, Effluents, Domestic Water Supplies, Leachates, Sewages,

Industrial Wastes

### SAMPLING:

Quantity Required

: 10 mL : Plastic

Container

# ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate (red), and the absorbance of the latter is measured colourimetrically.

Approximate absorbance: 0.5 at the full scale level.

## INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 cm light path

Data capture, reduction, and processing via a multistage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

## CALIBRATION:

BL plus 10 standards

### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

### NOTES:

This workstation was created Oct. 22/87 to take over all chloride testing being done on the ROM workstation, and at the separate "chloride only" sub-workstation. The original channels were retired at that time. The COCL workstation uses the identical method with a minor range change to suit the range of values expected from the full sample load. Chloride testing for river and lake samples collected under the APIOS program, and for precipitation samples, is performed by ion chromatography at the PRIC1 workstation.

## CHLORIDE

## QUALITY CONTROL DATA FROM 02/01/90 TO 28/12/90

Lab: Colourimetry

Analytical Range: - to 100 mg/L as Cl

# **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
<b>A</b> :	130	75.0	75.09	0.09	0.268
B :	130	25.0	24.99	-0.01	0.166
A+B:	130	100.0	100.08	0.08	0.355
A-B:	130	50.0	50.10	0.10	0.269
<b>C</b> :	130	25.0	24.99	-0.01	0.166
D:	130	5.0	4.97	-0.03	0.122
C+D:	130	30.0	29.96	-0.04	0.237
C-D:	130	20.0	20.02	0.02	0.168

s.d.(AB) S(between runs): 0.22

Sw(within run): 0.19 S/Sw: 1.17

s.d.(CD) S(between runs): 0.14

Sw(within run): 0.12 S/Sw: 1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

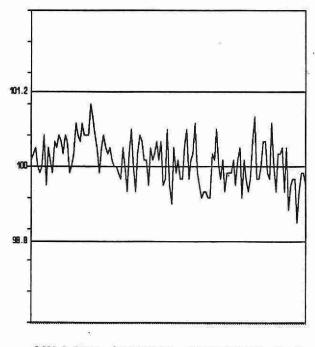
98.8 101.2 for A+B 49.2 50.8 for A-B 29.3 30.7 C+D for 19.55 20.45 for C-D

# **DUPLICATES:**

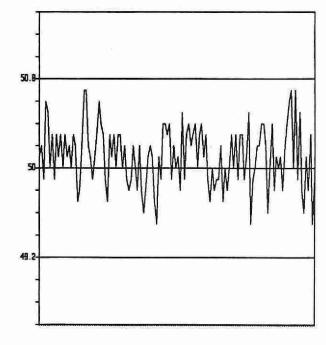
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*********				*************	
91	0.0	1.0	10.0	0.176	5.14
77	10.0		20.0	0.288	3.63
171	20.0	<b>H</b> 0)	100.0	0.364	1.26
339	Overall			0.292	on commonwe

	of Data	Data Mean	Standard(1) Deviation
		******	*********
Long Term Blank	130	0.015	0.255

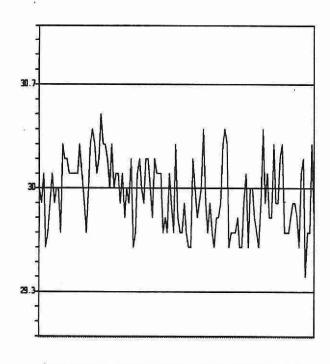
# QUALITY CONTROL DATA FROM 02/01/90 TO 28/12/90



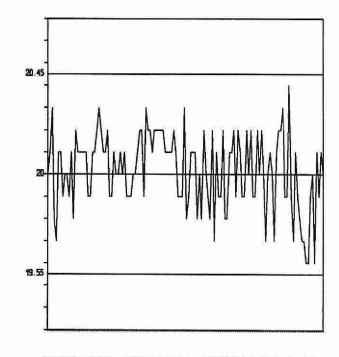
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## *** CHLORIDE ***

### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/78

LIS Test Name Code Work Station Code : CLIDUR : PRIC1

Unit Code

Units

: mg/L as Cl : 064960

Method Code

: 005AI0

Supervisor

: F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

### SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards.

Nitrogen-nitrate and sulphate are determined simultaneously.

### **INSTRUMENTATION:**

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

### NOTES:

Two analytical ranges are in operation at this work station, and subsequently, quality control results are provided for each range.

## CHLORIDE

# QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90

Lab: Ion Chromatography

Analytical Range: - to 1.0 mg/L as Cl

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
<b>A</b> :	44	0.8	0.804	0.004	0.018
B :	44	0.2	0.201	0.001	0.014
A+B:	44	1.0	1.005	0.004	0.024
A-B:	44	0.6	0.603	0.003	0.021

s.d.(AB) S(between runs): 0.016 Sw(within run): 0.015 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.95 1.05 for A+B 0.57 0.63 for A-B

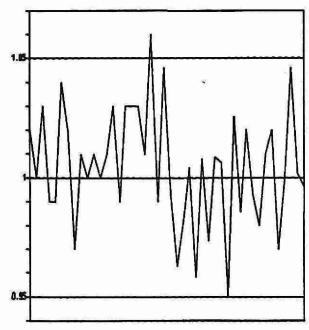
## **DUPLICATES:**

Number of Data Pairs	C	Samp oncn S		Mean(2) s.d.	Coefficient of var.(%)	
25	0.00		0.05	0.0115	35.0	
29	0.05	-	0.10	0.0149	19.9	
66	0.10	-	1.00	0.0149	6.6	
120	(	)veral	1	0.0142	7.7.5	

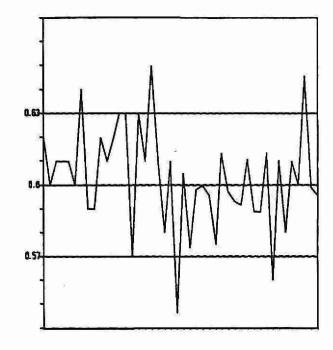
	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	44	0.009	0.012

CHLORIDE (mg/L as Cl)

# QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

_____ CONTROL LIMIT

## CHLORIDE

# QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90

Lab: Ion Chromatography

Analytical Range: - to 2.0 mg/L as Cl

# CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			********		
<b>A</b> :	90	1.60	1.599	-0.001	0.017
B :	90	0.40	0.398	-0.002	0.021
A+B:	90	2.00	1.997	-0.003	0.030
<b>A-B</b> :	90	1.20	1.201	0.001	0.024

s.d.(AB) S(between runs): 0.019 Sw(within run): 0.017 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 1.14 2.09 1.26 for A-B

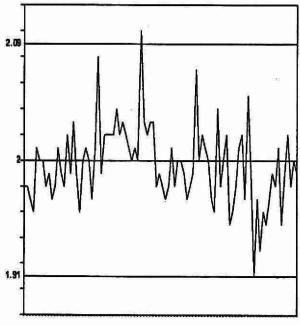
## **DUPLICATES:**

Number of Data Pairs	C	Samp oncn S		Mean(2) s.d.	Coefficient of var.(%)
23	0.00	-	0.20	0.0108	20.8
98	0.20	-	0.50	0.0110	1.4
61	0.50	-	1.00	0.0174	2.7
34	1.00	-	2.00	0.0228	2.7
216	(	)veral		0.0145	

	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	90	0.009	0.052

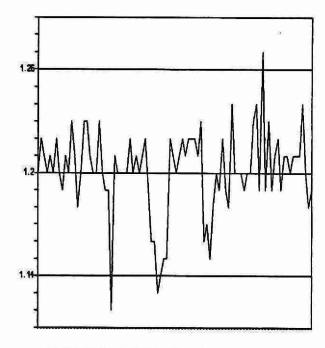
CHLORIDE (mg/L as C1)

# QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90





QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

_____ CONTROL LIMIT

### *** CHLORIDE ***

### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/78

LIS Test Name Code

: CLIDUR

Units

: ug/Filter as Cl

Work Station Code

: PRLOV

Unit Code

: 361960

Method Code

: 004AIC

Supervisor

: F. Lo

Sample Type/Matrix

: W40 filters from LoVol filter packs

### SAMPLING:

Quantity Required

: 1 filter

Container

: 50 mL polypropylene tube

### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample peak heights to a series of standards. Results are converted to ug/filter as Cl.

Nitrogen-nitrate and sulphate are determined simultaneously.

#### INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

### CALIBRATION:

BL plus 9 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

## NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

## **CHLORIDE**

# QUALITY CONTROL DATA FROM 19/01/90 TO 20/12/90

Lab: Ion Chromatography

Analytical Range: - to 100 µg/filter as Cl

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			*******		
A :	24	80.0	80.13	0.13	0.97
B :	24	20.0	20.06	0.06	0.74
A+B:	24	100.0	100.19	0.19	1.20
A-B:	24	60.0	60.06	0.06	1.25

s.d.(AB) S(between runs): 0.86

Sw(within run): 0.88 S/Sw: 0.98

On any given day the calibration is accepted if the values obtained lie within the ranges:

96.3 57.5

103.7

for A+B

62.5

for A-B

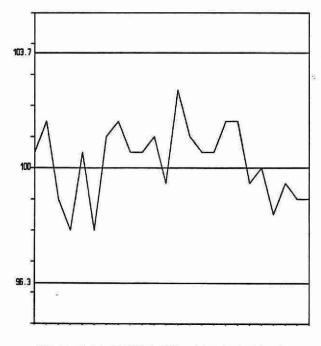
# **DUPLICATES:**

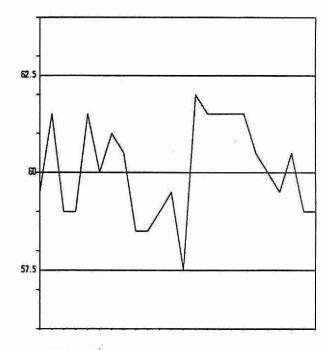
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
7	0.0	-	10.0	0.105	1.6
12	10.0	=	20.0	0.319	2.3
10	20.0	-	100.0	1.416	1.9
29	(	Overal	1	0.249	

	Number of Data	Data Mean	Standard(1) Deviation	
Long Term Blank	24	0.042	0,204	

CHLORIDE (ug/filter as Cl)

QUALITY CONTROL DATA FROM 19/01/90 TO 20/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

## *** CHLOROPHYLL ***

### IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code

Method Code

Sample Type/Matrix

: BOD

: CHLRAT, CHLRAC, CHLRBT, Units : RCHLO Unit Code

: 002DS2

: Rivers, Lakes, Effluents

Method Introduced

Supervisor

: 01/04/75 : ug/L

: 063000

: P. Campbell

## SAMPLING:

Quantity Required

Container

: 1000 mL for clear samples; 500 mL if visibly green

: Glass or plastic

Other

: In the field a sample is filtered through a nylon filter. The filter is folded and then placed between two membrane filter-support pads, and the package is enclosed in a plastic dish labelled with the sample number and sample volume filtered, the dish is kept in the dark or wrapped in aluminum foil, and shipped immediately, or kept

frozen.

### ANALYTICAL PROCEDURE:

Using a Commodore PET microcomputer-controlled, automated spectrophotometer, two scans are developed with absorbance measurements at 630, 645, and 663 nm for the first scans; the minimum absorbance value between 710 and 750 nm (readings at 5 nm intervals) is utilized as a turbidity correction. Chlorophyll "a" and "b" are calculated from this scan. After automated acidification, the second scan is obtained from the wavelength 665 nm for correcting chlorophyll "a" measurement. SCOR-UNESCO equations are used for all chlorophyll calculations.

#### INSTRUMENTATION:

-Automated modular continuous flow scanning spectrophotometer system

-Microcomputer system for control of sampling, timing and data processing (i.e. data capture, calculations and transfer of results to LIS)

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2, 0.2, 0.1*

T value: 1, 1, 0.5°

### CONTROLS:

Calibration

: LTBL plus 2 "standards", e.g. QCA

Drift

: "standard", BL every 20 samples

### NOTES:

"Standards" are prepared from chlorophyll "a" and "b", but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages.

* Chlorophyll "a", "a" acidified (corrected), and "b" respectively.

# CHLOROPHYLL "a"

# QUALITY CONTROL DATA FROM 01/01/90 TO 30/11/90

Lab: BOD

Analytical Range: - to 50 ug/L

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	136	3.0	2.98	-0.02	0.1402
B :	136	1.0	1.01	0.01	0.0708
A+B:	136	4.0	3.98	-0.02	0.1901
A-B:	136	2.0	1.97	-0.03	0.1149

s.d.(AB) S(between runs): 0.11 Sw(within run): 0.08 S/Sw: 1.4

On any given day the calibration is accepted if the values obtained lie within the ranges:

3.6 1.8 4.4 2.2 for A-B

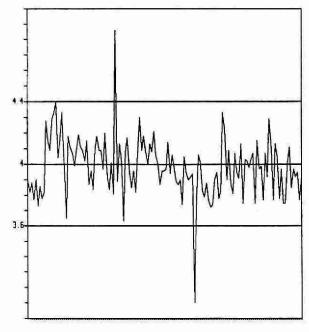
# **DUPLICATES:**

Number of Data Pairs			Mean(2) s.d.	Coefficient of var.(%)	
	~~~~	******			
149	0.0		5.0	0.2065	8.3
58	5.0	-1	25.0	0.6928	7.7
9	25.0		50.0	2.6456	6.6
216	(Overal!	l	0.3286	

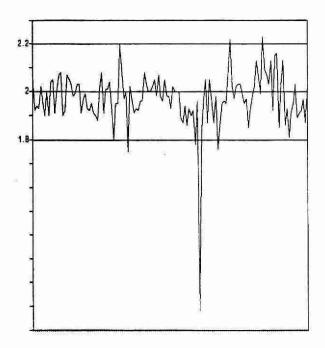
	Number	Data	Standard(1)
	of Data	Mean	Deviation
Blank	136	0.049	0.0463

CHLOROPHYLL "a" (ug/L)

QUALITY CONTROL DATA FROM 01/01/90 TO 30/11/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

___ CONTROL LIMIT

CHLOROPHYLL "a", ACIDIFIED

QUALITY CONTROL DATA FROM 01/01/90 TO 30/11/90

Lab: BOD

Analytical Range: - to 10.0 ug/L

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	109	2.4	2.46	0.06	0.212
B :	109	0.8	0.78	-0.02	0.125
A+B:	109	3.2	3.24	0.04	0.282
A-B:	109	1.6	1.68	0.08	0.204

s.d.(AB) S(between runs): 0.18 Sw(within run): 0.14 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.40 1.10 4.00 2.10 for for A-B

DUPLICATES:

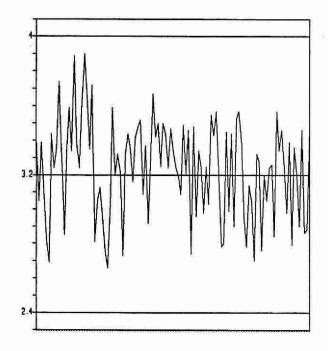
Number of Data Pairs				Mean(2) s.d.	Coefficient of var.(%)

11	0.0		3.0	0.1740	7.5
12	3.0	-	5.0	0.3167	8.7
18	5.0	-	10.0	0.3486	7.1
41	i	Overal		0.3168	***

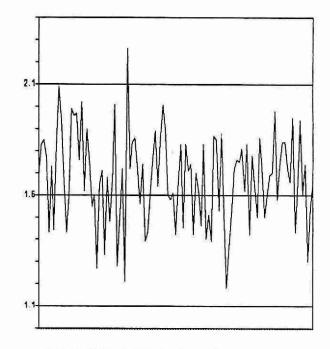
	Number of Data	Data Mean	Standard(1) Deviation	
	(*************************************			
Blank	109	-0.043	0.145	

CHLOROPHYLL "a", ACIDIFIED (ug/L)

QUALITY CONTROL DATA FROM 01/01/90 TO 30/11/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CHLOROPHYLL "b"

QUALITY CONTROL DATA FROM 01/01/90 TO 30/11/90

Lab: BOD

Analytical Range: - to 1.0 ug/L

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	136	3.0	2.928	-0.072	0.1334
B :	136	1.0	1.008	0.008	0.0908
A+B:	136	4.0	3.936	-0.064	0.1933
A-B:	136	2.0	1.920	-0.080	0.1214

s.d.(AB) S(between runs): 0.11 Sw(within run): 0.09 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

3.60 for 1.80 2.20 for A-B

DUPLICATES:

Number of Data Pairs	C	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)

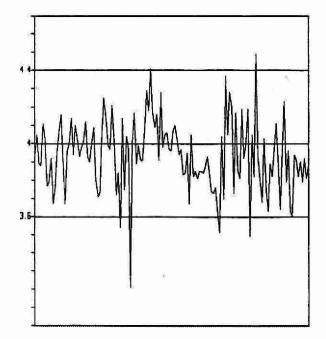
73	0.00	-	0.50	0.0814	20.4
109	0.50	*	2.00	0.1445	16.1
31	2.00	-	10.00	0.3309	4.6
213	. (Overall		0.1395	

	Number of Data	Data Mean	Standard(1) Deviation

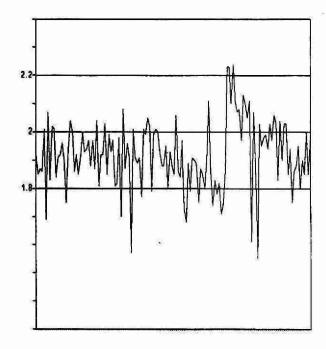
Blank	136	0.057	0.069

CHLOROPHYLL "b" (ug/L)

QUALITY CONTROL DATA FROM 01/01/90 TO 30/11/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

*** CLAY ***

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: CLAY

Units

: % by weight

Work Station Code Method Code : DOPARTSZ : AM1002 Unit Code Supervisor : 070000 : A. Neary

Sample Type/Matrix

: Soil

SAMPLING:

Quantity Required

: 20 g dry : Glass

Container

. 0.000

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

To prevent flocculation a portion of sample, pretreated for organic matter and carbonate removal, is dispersed in a sodium hexametaphosphate solution. The sand fraction (>53 um) is removed by wet sieving; the silt and clay fraction is dispersed in a sedimentation cylinder. Measurement of the percent silt and clay in the sample is based on the settling velocities of spherical particles by the application of Stokes Law.

INSTRUMENTATION:

-Sartorius 4 place digital balance (Handy)

-Balance accurate to 0.0001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 1

T value: 5

CALIBRATION:

Balance zero

CONTROLS:

Recovery:

2 long term soil samples representing different soil types plus round robin ECSS samples

(run occasionally)

CLAY

QUALITY CONTROL DATA FROM 01/06/90 TO 22/11/90

Lab: Dorset Soils

Analytical Range: - to 100 % by wt.

RECOVERIES:

Number of Data		Av. Concn Measured	Standard(1) Deviation	

R1:	10	53.5	1.958	
R2:	9	1.11	1.167	

DUPLICATES:

Number of	C	Sam		Mean(2)	Coefficient
Data Pairs	<u></u>	onen	Span	s.d.	of var.(%)
10	0.0	-	20.0	1.190	39.4
1	20.0	-5	50.0	N.A.	N.A.
2	50.0	-	100.0	N.A.	N.A.
12	(Overa	all	1.178	

*** COLOUR, TRUE ***

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code Method Code Sample Type/Matrix

: Dorset : COLTR

: DOCC : 1102KP

: Streams, Lakes

Method Introduced

Units

Unit Code

Supervisor

: 15/10/80 : TCU : 340000

: A. Neary

SAMPLING:

Quantity Required

: 75 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured on a settled sample colourimetrically in a system calibrated with acidified chloroplatinate standards. Colour is measured using a 400-450 nm broadband blue filter. Turbidity effects are partially suppressed by using a broadband red filter. True colour is calculated from the two absorbance measurements using an empirically derived equation.

Approximate absorbance: 0.05 at the full scale level.

INSTRUMENTATION:

Two colourimeters, one with broadband blue filter (400-450 nm) and the other with broadband red filter (660-740 nm). Colourimetric measurement is through a 4.0 cm. light path.

REPORTING:

Maximum Significant Figures: 3

Current W value: 1.0

T value: 5

CALIBRATION:

Blank Only

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA, QCB

NOTES:

Slope factor is changed whenever light source in a colourimeter or cell is replaced. This is accomplished by analyzing 7 standards.

COLOUR, TRUE

QUALITY CONTROL DATA FROM 11/01/90 TO 20/12/90

Lab: Dorset

Analytical Range: - to 100 TCU

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			*************		*************
A :	83	50.0	48.64	-1.36	2.85
B :	83	10.0	9.39	-0.61	1.73
A+B:	83	60.0	57.99	-2.01	3.87
A-B:	83	40.0	39.21	-0.79	2.67

s.d.(AB) S(between runs): 3.0

Sw(within run): 1.9 S/Sw: 1.6

On any given day the calibration is accepted if the values obtained lie within the ranges:

53 - 67 for A+B 35 - 45 for A-B

DUPLICATES:

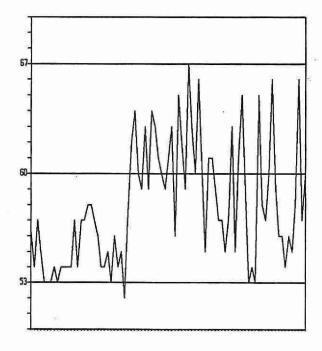
Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)

68	0.0	÷	20.0	1.36	23.6
59	20.0	-	50.0	1.66	6.6
58	50.0		100.0	2.48	4.3
185		Overa:	1	1.84	

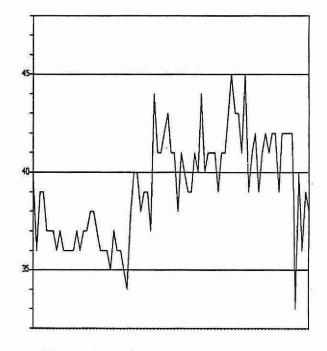
	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	83	0.018	0.165

COLOUR, TRUE (TCU)

QUALITY CONTROL DATA FROM 11/01/90 TO 20/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

*** COLOUR, TRUE ***

IDENTIFICATION:

Laboratory

: Colourimetry

Method Introduced

: 13/03/84

LIS Test Name Code Work Station Code

: COLTR : WCOL

Unit Code

Units

: TCU : 340000

Method Code Sample Type/Matrix : 102BC9 : Domestic Waters, Effluents, Surface Waters, Industrial Wastes, Leachates

Supervisor

: M. Rawlings

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference

Approximate absorbance: 0.3 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450 nm). Turbidity measurement is through a 5.0 cm, light path using a different broadband filter (660-740 nm). Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

COLOUR, TRUE

QUALITY CONTROL DATA FROM 03/01/90 TO 21/12/90

Lab: Colourimetry

Analytical Range: - to 100 TCU

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
			***********	***************************************	**********
A :	74	70.0	70.36	0.34	0.470
B :	74	25.0	24.44	-0.56	0.453
A+B:	74	95.0	94.80	-0.20	0.744
A-B:	74	45.0	45.93	0.93	0.547
C :	74	25.0	24.44	-0.56	0.453
D:	74	7.5	7.40	-0.10	0.270
C+D:	74	32.5	31.84	-0.66	0.584
C-D:	74	17.5	17.03	-0.47	0.463

s.d.(AB) S(between runs): 0.46

Sw(within run): 0.39 S/Sw: 1.19

s.d.(CD) S(between runs): 0.37

Sw(within run): 0.33 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

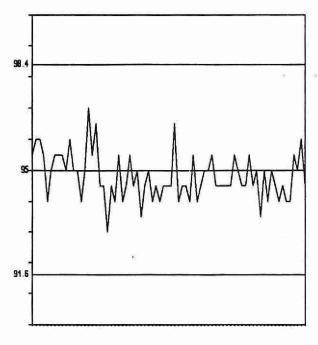
91.60 - 98.40 for A+B 42.75 - 47.25 for A-B 29.60 - 35.40 for C+D 15.55 - 19.45 for C-D

DUPLICATES:

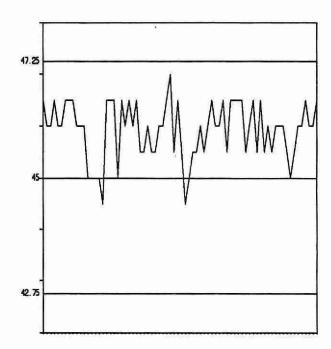
Number of Data Pairs			Mean(2) s.d.	Coefficient of var.(%)	

124	0.00		5.00	0.353	19.13
63	5.00		25.00	0.398	5.38
22	25.00	J#	100.00	0.828	0.84
209	(Overal		0.392	

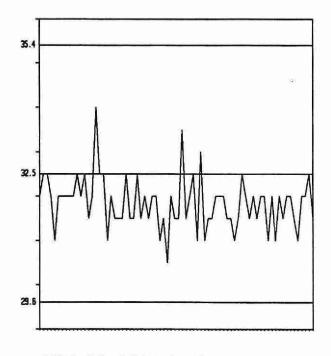
	Number of Data	Data Mean	Standard(1) Deviation
			77777777777
Long Term Blank	74	0.094	0.283



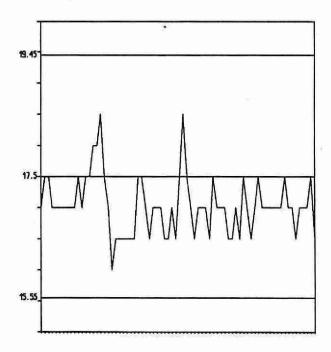
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 01/06/76

LIS Test Name Code

: COND25

Units
Unit Code

: uS/cm at 25°C

Work Station Code Method Code : DOCC : 0903CM Unit Code Supervisor : 350351 : A. Neary

Sample Type/Matrix

: Streams, Lakes, Precipitation, Soil Leachates

SAMPLING:

Quantity Required

: 75 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell and equilibrated to 25°C. The conductivity is read directly from a digital display.

INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

None

CONTROLS:

Calibration

: LTB plus 4 standards, e.g. QCA, QCB, 147 uS/cm plus 717.7 uS/cm stds.

NOTES:

*T value is based on duplicate analyses at concentrations above the lowest range.

CONDUCTIVITY

QUALITY CONTROL DATA FROM 06/04/90 TO 20/12/90

Lab: Dorset

Analytical Range: - to 300 uS/cm

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	88	112.0	111.88	0.12	0.611
B :	88	23.5	23.64	0.14	0.416
A+B:	88	135.5	135.53	0.03	0.841
A-B:	88	88.5	88.25	-0.25	0.621

s.d.(AB) S(between runs): 0.52

Sw(within run): 0.44 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

133 - 138 for A+B 90.4 - 86.6 for A-B

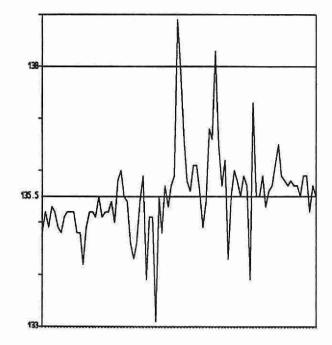
DUPLICATES:

Number of San Data Pairs Concr				Mean(2) s.d.	Coefficient of var.(%)
22	0.0	e le	20.0	0.248	2.1
207	20.0		100.0	0.271	1.1
13	100.0	-	300.0	0.755	0.5
242	(Overal	11	0.292	735

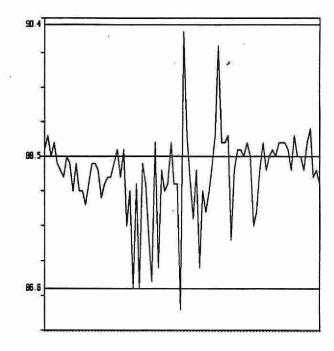
	Number of Data	Data Mean	Standard(1) Deviation
	******	**********	
Long Term Blank	88	1.25	0.342

CONDUCTIVITY (us/cm)

QUALITY CONTROL DATA FROM 06/04/90 TO 20/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/78

LIS Test Name Code Work Station Code : COND25 : PRCON Units Unit Code : uS/cm at 25°C : 350351

Method Code

: 002BI2

Supervisor

: 350351 : F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

After equilibration at 25°C, The conductivity of the sample is measured.

INSTRUMENTATION:

Automated modular continuous flow conductivity system comprised of sampler, water bath, conductivity meter with cell, chart recorder.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

Compatibility between conductivity meter and chart recorder is confirmed by checking 3 standard resistances.

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 solution every 10 samples

NOTES:

A calibration standard for the ion chromatographic system is used to monitor the drift for the conductivity system, but its theoretical conductivity is unknown.

CONDUCTIVITY

QUALITY CONTROL DATA FROM 15/01/90 TO 18/12/90

Lab: Ion Chromatography

Analytical Range: - to 100.0 μS/cm

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
				*********	***********
A :	39	44.5	45.8	1.3	1.193
B:	39	7.5	8.2	0.7	0.550
A+B:	39	52.0	54.0	2.0	1.488
A-B :	39	37.0	37.5	0.5	1.112

s.d.(AB) S(between runs): 0.93 Sw(within run): 0.79 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

43.0 31.0 61.0 for A+B 43.0 for A-B

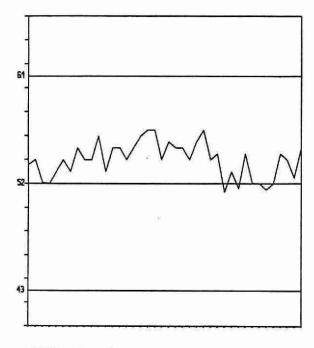
DUPLICATES:

Number of Data Pairs		Samp Concn S	Span	Mean(2) s.d.	Coefficient of var.(%)
18	0.0	_	10.0	0.3909	7.1
44	10.0	-	25.0	0.9245	5.0
35	25.0	+	50.0	1.2895	4.1
9	50.0	+	100.0	2.2219	3.5
106		Overal	l	1.0448	

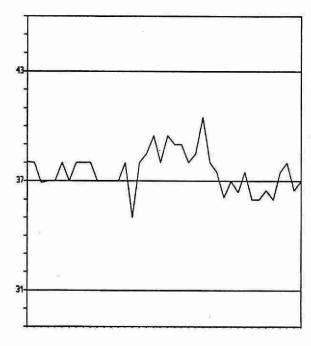
	Number of Data	Data Mean	Standard(1) Deviation
			allo 400 400 ani ne
Long Term Blank	39	0.4846	0.0670

CONDUCTIVITY (us/cm)

QUALITY CONTROL DATA FROM 15/01/90 TO 18/12/90







QUALITY CONTROL STANDARD A-B

____ CONTROL LIMIT

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: 01/04/74

LIS Test Name Code

: COND25

Units

: uS/cm at 25°C

Work Station Code

: RATS

Unit Code

: 350351

Method Code Sample Type/Matrix : 002B12 : Rivers, Lakes Supervisor

: F. Lo

SAMPLING:

Quantity Required

: 25 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured. pH, Gran alkalinity and total fixed endpoint alkalinity are determined simultaneously.

INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler, water bath, pump, conductivity meter with cell plus microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

CONTROLS:

Calibration

: BL plus 3 standards, e.g. QCA

Drift

: In run standards throughout the run (tap water diluted to 20% V/V)

CONDUCTIVITY

QUALITY CONTROL DATA FROM 03/01/90 TO 27/12/90

Lab: Titration

Analytical Range: - to 2000 uS/cm

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	********	********		************	
A :	79	717.8	716.4	-1.4	2.357
B :	79	147.0	147.6	0.6	0.692
A+B:	79	864.8	864.0	-0.8	2.519
A-B:	79	570.8	568.8	-1.2	2.392
C :	79	147.0	147.6	0.6	0.692
D:	79	37.1	38.1	1.0	0.362
C+D:	79	184.1	185.7	1.6	0.823
C-D:	79	109.9	109.4	-0.5	0.737

s.d.(AB) S(between runs): 1.74 S

Sw(within run): 1.69 S/Sw: 1.03

s.d.(CD) S(between runs): 0.55

Sw(within run): 0.52 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

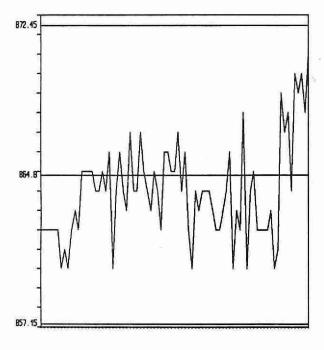
857.15 872.45 for A+B 565.7 575.9 for A-B 180.1 188.11 for C+D 107.23 112.57 for C-D

DUPLICATES:

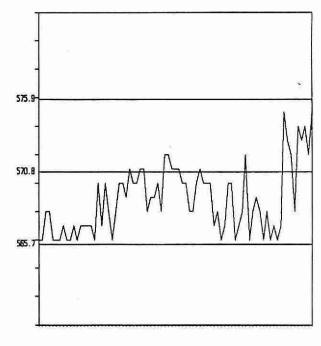
Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
59	0	•	200	2.014	2.8
72	200 -	•	500	2.160	0.6
89	500		1000	3.375	0.6
2	1000	3	2000	N.A	N.A
222	Overall			2.600	

CONDUCTIVITY (us/cm)

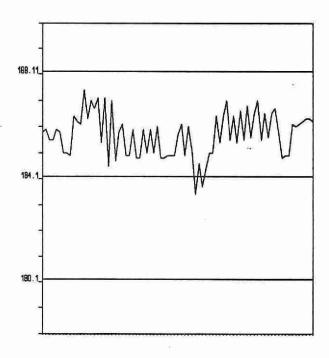
QUALITY CONTROL DATA FROM 03/01/90 TO 27/12/90



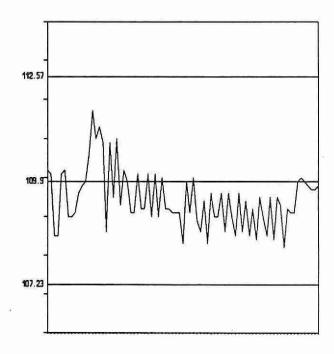
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory: Titration: Method Introduced: 01/04/74

LIS Test Name Code : COND25 Units : uS/cm at 25°C

Work Station Code : WATS Unit Code : 350351
Method Code : 002BI2 Supervisor : F. Lo

Sample Type/Matrix : Domestic Waters, Sewage, Industrial effluents

SAMPLING:

Quantity Required : 25 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured. pH and Total fixed endpoint alkalinity are determined simultaneously.

INSTRUMENTATION:

Automated modular continual flow conductivity system comprising of a sampler, water bath, pump, conductivity meter with cell plus microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3 Current W value: 1 T value: 5

CONTROLS:

Calibration : LTBL plus 3 standards, e.g. QCA

Drift: In run standards throughout the run (tap water diluted to 50% V/V)

CONDUCTIVITY

QUALITY CONTROL DATA FROM 02/01/90 TO 31/12/90

Lab: Titration

Analytical Range: - to 2000 uS/cm

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	OI Data	Conci		Dias	Deviation
A:	164	1413.0	1410	-3.0	5.311
B :	164	717.8	716	-1.8	3.157
A+B:	164	2130.8	2126	-4.8	7.423
A-B:	164	695.2	694	-1.2	4.609
C :	164	717.8	716	-1.8	3.157
D:	164	147.0	149	2.0	1.038
C+D:	164	864.8	865	0.2	3.736
C-D:	164	570.8	567	-3.8	2.853

s.d.(AB) S(between runs): 4.37 Sw(within run): 3.26 S/Sw: 1.3

s.d.(CD) S(between runs): 2.35 Sw(within run): 2.02 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

2106.05 2155.55 for A+B 678.70 711.70 for A-B 851.48 878.12 for C+D 561.92 579.68 C-D for

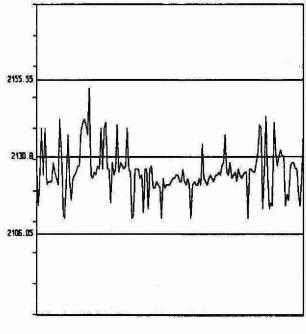
DUPLICATES:

Number of Data Pairs	364 A	Samı Concn		Mean(2) s.d.	Coefficient of var.(%)

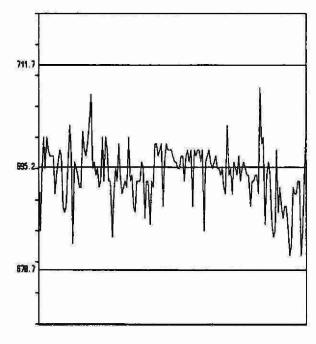
15	0	-	100	1.342	2.1
36	100	-	200	1.587	1.1
137	200	-	500	3.085	0.9
154	500	4	1000	6.193	1.1
40	1000	-	2000	14.020	1.0
382		Overal	1	4.882	2.2

CONDUCTIVITY (us/cm)

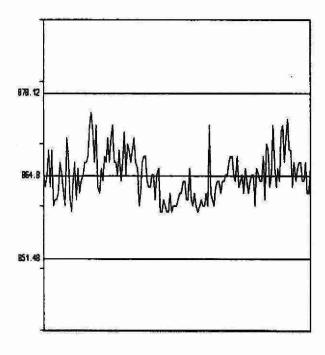
QUALITY CONTROL DATA FROM 02/01/90 TO 31/12/90



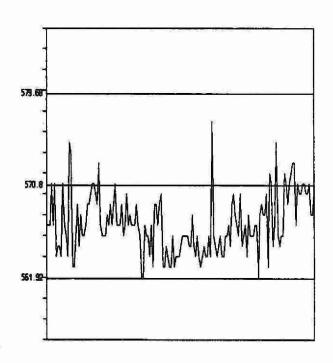
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

*** CONDUCTIVITY ***

IDENTIFICATION:

Laboratory

:

: Titration

Method Introduced

: 20/05/87

LIS Test Name Code Work Station Code

: COND25 : WQSDIRT Units Unit Code : uS/cm at 25°C

Method Code

: 004AB4

Unit Code

: 350351

Sample Type/Matrix

: Landfill leachates

Supervisor : F. Lo

SAMPLING:

Quantity Required

: 75 mL

Container

: Plastic or glass

ANALYTICAL PROCEDURE:

After equilibration at 25°C, the conductivity of the sample is measured; samples are filtered first if necessary. Analysis is performed on supernatant or filtrate.

INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 5

T value: 25

CONTROLS:

Calibration

: BL plus 4 standards, e.g. QCA

CONDUCTIVITY

QUALITY CONTROL DATA FROM 08/01/90 TO 17/12/90

Lab: Titration

Analytical Range: - to 10000 uS/cm

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	39	6668	6620	-48	22.41
B :	39	2767	2740	-27	13.08
A+B:	39	9435	9360	-75	28.83
A-B :	39	3901	3881	-20	22.69
C :	39	1413	1409	-4	6.05
D :	39	717	712	-5	2.84
C+D:	39	2130	2122	-8	7.47
C-D:	39	696	697	1	5.78

s.d.(AB) S(between runs): 18.3

Sw(within run): 16.0 S/Sw: 1.1

s.d.(CD) S(between runs): 4.7

Sw(within run): 4.1 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

9295 - 9575 for A+B 3808 - 3994 for A-B 2089 - 2171 for C+D 668 - 724 for C-D

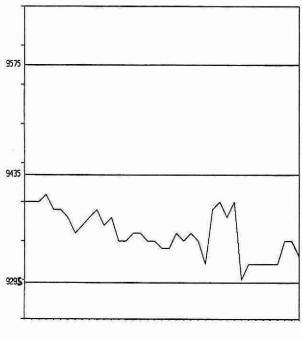
DUPLICATES:

Number of Sample Data Pairs Concn Span			Mean(2) s.d.	Coefficient of var.(%)	

19	0	•	500	2.090	0.9
28	500	3=0	1000	4.509	0.8
11	1000	-	10000	21.790	1.9
58		Over	all	5.148	, record

CONDUCTIVITY (us/cm)

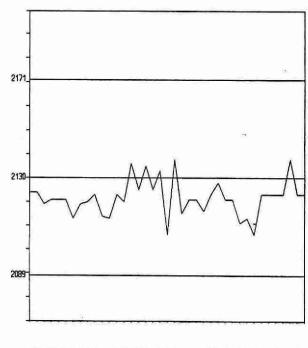
QUALITY CONTROL DATA FROM 08/01/90 TO 17/12/90

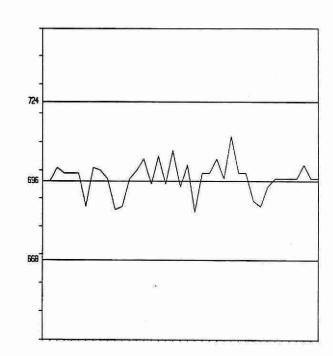


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QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B





QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

___ CONTROL LIMIT

*** COPPER, ACID EXTRACTABLE ***

IDENTIFICATION:

Laboratory LIS Test Name Code : Dorset Soils : CUUT

Method Introduced Units : 01/06/80 : ug/g as Cu

Work Station Code Method Code : DOHMTE : 551AA1

Unit Code Supervisor : 073829

Sample Type/Matrix

: Soil

visor : A. Neary

SAMPLING:

Quantity Required

: 1 g dry : Glass

Container

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm. A subsample is ground to less than 500 um (35 mesh).

ANALYTICAL PROCEDURE:

A 0.500 g sample plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125°C for 2 hours. The temperature is increased to 175°C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Cu by AAS at 324.8 nm using an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale value.

Lead, nickel and zinc are also determined on the same extract.

INSTRUMENTATION:

-Varian AA1275 with programmable sample changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.2

T value: 1.0

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration

: Three long term soil samples representing different soil types, 2 method blanks and one

judiciously blended sample extract run with each run.

Drift

: BL plus 1 standard (100% F.S.) every 10 samples

NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal. Values for recoveries are unknown - average value used.

COPPER, ACID-EXTRACTABLE

QUALITY CONTROL DATA FROM 30/03/90 TO 22/11/90

Lab: Dorset Soils

Analytical Range: - to 50.0 ug/g

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		***************************************		***************************************	
A :	5	37.0	38.14	1.14	0.626
B :	5	13.0	13.36	0.36	0.532
A+B:	5	50.0	51.50	1.50	0.659
A-B:	5	24.0	24.78	0.78	0.957

s.d.(AB) S(between runs): 0.58

Sw(within run): 0.68 S/Sw: 0.85

On any given day the calibration is accepted if the values obtained lie within the ranges:

42.5 - 57.5 for A+B 19.0 - 29.0 for A-B

RECOVERIES:

	Number of Data	Av. Concn Measured	Standard(1) Deviation
		•	
R1:	5	13.46	0.792
R2:	5	15.16	0.577
R3:	5	13.26	0.677

DUPLICATES:

Number of Data Pairs		Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
~~~~~~~					********	
10		0.0	( <del>100</del> 1)	10.0	0.829	10.7
3	*9	10.0	il <del>e</del> e	20.0	0.817	1.0
2		20.0	-	50.0	N.A	N.A
15		(	Overal	1	0.64	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
			•••••
Digested Blank	5	0.000	0.000

### *** COPPER, TOTAL ***

## **IDENTIFICATION:**

Laboratory

: Dorset

Method Introduced

: 01/03/86 : ug/L as Cu

LIS Test Name Code Work Station Code Method Code

: CUUT : DOASV Units Unit Code

: 063882

Sample Type/Matrix

: 001PP2

Supervisor

: A. Neary

: Streams, Lakes, Precipitation

### SAMPLING:

Quantity Required

: 100 mL

Container

: 500 mL, acid washed Nalgene Teflon container, bagged in a clean room

## ANALYTICAL PROCEDURE:

Samples are acidified to 0.1% using Seastar nitric acid in a clean room. Oxygen is removed by nitrogen gas and samples are analyzed using anodic stripping voltammetry on a hanging mercury drop electrode. Change in current when copper is stripped from mercury drop is proportional to concentration.

#### **INSTRUMENTATION:**

Metrohm 646 VA Processor with Model 675 VA Sample Changer

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.3

T value: 1.5

### CALIBRATION:

BL plus 2 standards daily

#### CONTROL:

Calibration

: BL plus 2 standards, e.g. QCA and EPA standard

Drift

: End of every run (approximately every 8 samples)

## COPPER, TOTAL

## QUALITY CONTROL DATA FROM 09/01/90 TO 18/12/90

Lab: Dorset Soils

Analytical Range: - to 4.00 ug/L as Cu

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
					***********
A :	42	3.60	3.91	0.31	0.449
B :	42	0.90	1.17	0.27	0.291
A+B:	42	4.50	5.08	0.58	0.513
A-B:	42	2.70	2.74	0.04	0.555

s.d.(AB) S(between runs): 0.38

Sw(within run): 0.39 S/Sw: 0.96

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.78 - 7.22 for A+B 0.66 - 4.74 for A-B

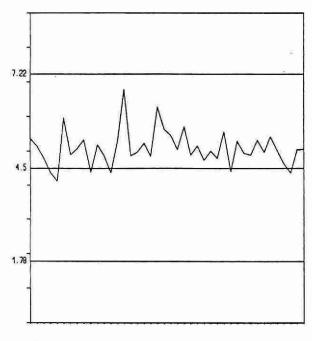
## **DUPLICATES:**

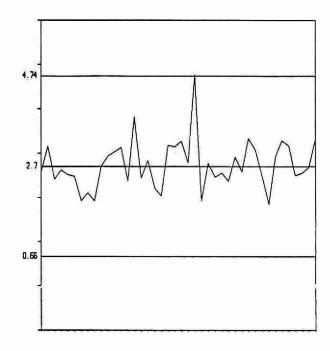
Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
6	0.00	i <del>lli</del>	0.50	0.147	36.5
10	0.50	784	1.00	0.191	21.8
8	1.00	-	4.00	0.269	13.8
24	O	veral	I	0.211	

	Number of Data	Data Mean	Standard(1) Deviation	
Long Term Blank	42	0.112	0.265	

COPPER, TOTAL (ug/L as Cu)

## QUALITY CONTROL DATA FROM 09/01/90 TO 18/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

-

CONTROL LIMIT

## *** FLUORIDE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code Work Station Code : FFIDUR : DOSPF Units Unit Code : ug/L as F : 063809

Method Code Sample Type/Matrix : 001AIE S : Precipitation, Lakes, and Streams

Supervisor : A. Neary

### SAMPLING:

Quantity Required

: 50 mL

Container

: Plastic

### ANALYTICAL PROCEDURE:

Fluoride is determined via an automated flow system for which the detector is a specific ion electrode; prior to measurement the sample is mixed with a high ionic strength buffer containing; sodium citrate, disodium ethylenediaminetetraacetate (EDTA), phosphoric acid, and sufficient sodium hydroxide to obtain pH 6.7.

#### INSTRUMENTATION:

Automated modular continuous flow ion specific electrode system.

### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.2

T value: 1

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: 2 standards, e.g. QCA

Drift

: BL plus 1 standard in duplicate

Interference

: Combined fluoride and aluminum standard confirms that aluminum is not an

interference.

#### NOTES:

Values for recoveries are based upon the average recovery value obtained.

## **FLUORIDE**

## QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90

Lab: Dorset Soils

Analytical Range: - to 70.0 ug/L as F

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
				********	*******
A :	69	48	47.6	-0.4	0.706
B :	69	24	23.7	-0.3	0.491
A+B:	69	72	71.3	-0.7	1.000
<b>A-B</b> :	69	24	23.9	-0.1	0.694

s.d.(AB) S(between runs): 0.61

Sw(within run): 0.49 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

67.5 - 76.5 for A+B 21.0 - 27.0 for A-B

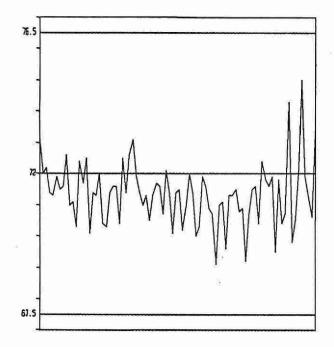
## **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
	\ <del></del>				
26	0.0		10.0	0.409	6.3
26 134	10.0		40.0	0.601	2.5
127	40.0	-	70.0	0.859	1.7
287		Overal	ľ	0.742	

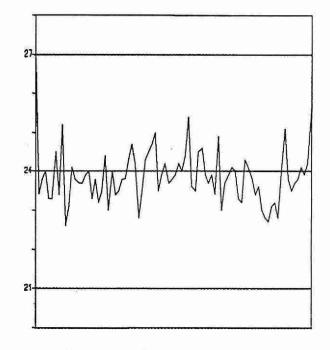
	Number of Data	Data Mean	Standard(1) Deviation
Al Interference	84	59.92	1.223

# FLUORIDE (ug/L as F)

## QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90







QUALITY CONTROL SAMPLE A-B

CONTROL LIMIT

### *** FLUORIDE ***

#### **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: Before '74

LIS Test Name Code

: FFIDUR

Units Unit Code : mg/L as F : 064809

Work Station Code Method Code

: WFNO3 : 003AC2

Supervisor

: M. Rawlings

Sample Type/Matrix

: Domestic Waters, Surface Waters, Leachates, Effluents

### SAMPLING:

Quantity Required

: 50 mL

Container

: Plastic

## ANALYTICAL PROCEDURE:

Using an automated flow system the sample is distilled in the presence of sulphuric acid at 160°C; the distillate is then reacted (in an acetic acid-acetate buffer media) with Alizarin Fluorine Blue and lanthanum nitrate to form a ternary Alizarin Blue-lanthanide-fluoride complex.

Approximate absorbance: 0.8 at the full scale level.

#### **INSTRUMENTATION:**

Modular continuous flow colourimetric system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture, reduction, and processing via a muli-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 6 standards

### CONTROLS:

Calibration

: 2 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

### FLUORIDE

## QUALITY CONTROL DATA FROM 16/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 2.0 mg/L as F

### CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	105	1.6	1.604	0.004	0.0138
B :	105	0.8	0.803	0.003	0.0109
A+B:	105	2.4	2.408	0.008	0.0178
A-B:	105	0.8	0.801	0.001	0.0173
C :	105	0.8	0.803	0.003	0.0109
D:	105	0.16	0.160	0.000	0.0068
C+D:	105	0.96	0.963	0.003	0.0138
C-D:	105	0.64	0.644	0.004	0.0118

s.d.(AB) S(between runs): 0.013

Sw(within run): 0.012 S/Sw: 1.02

s.d.(CD) S(between runs): 0.009

Sw(within run): 0.008 S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.28		2.52	for	A+B
0.72		0.88	for	A-B
0.91		1.01	for	C+D
0.59	-	0.69	for	C-D

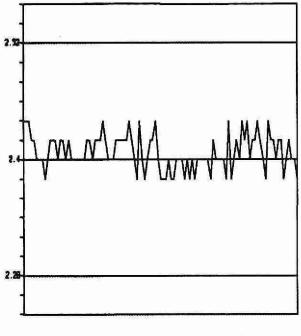
### **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*****				**********	
250	0.00		0.40	0.0150	15.0
29	0.40	100	1.00	0.0134	1.7
26	1.00	-	2.00	0.0163	1.2
305	C	)verall		0.0148	

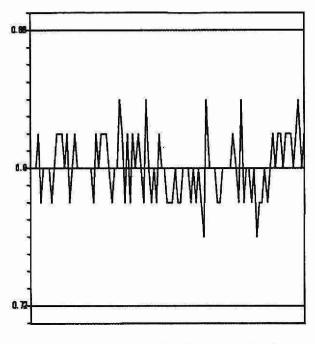
	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	105	-0.00095	0.0075

## FLUORIDE (mg/L as F)

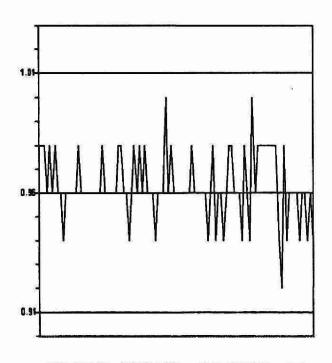
## QUALITY CONTROL DATA FROM 16/01/90 TO 20/12/90



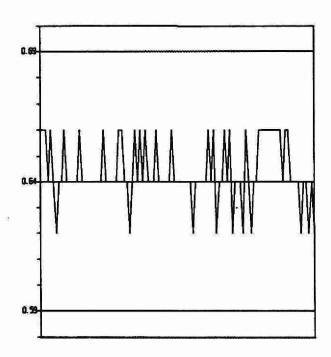
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

#### *** HARDNESS ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 01/04/74

LIS Test Name Code

: HARDT : RMAAS Units Unit Code : mg/L as CaCO₃

Work Station Code Method Code

: RMAAS

Supervisor

: 064915 : M. Young

Sample Type/Matrix

: Rivers, Lakes, Soil Extracts

### SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

#### ANALYTICAL PROCEDURE:

Samples are analysed for calcium and magnesium by AAS at workstation RMAAS. Hardness is calculated using the formula:

HARDT = (CAUR * 2.497) + (MGUR * 4.116)

#### **INSTRUMENTATION:**

Automated flow injection atomic absorption spetrophotometer (AAS) system.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1.0

#### CALIBRATION:

Refer to Calcium and Magnesium tests at RMAAS

#### CONTROLS:

Refer to Calcium and Magnesium tests at RMAAS

### *** HARDNESS ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 08/04/86

LIS Test Name Code Work Station Code : HARDT : WAAS

Unit Code

Units

: mg/L as CaCO₃ : 064915

Method Code

: CALC01

Supervisor

: M. Young

Sample Type/Matrix

: Domestic Waters, Leachates, Effluents, Sewage, Industrial Wastes

### SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

#### ANALYTICAL PROCEDURE:

Samples are analysed for calcium and magnesium by AAS at workstation WAAS. Hardness is calculated using the formula:

HARDT = (CAUR * 2.497) + (MGUR * 4.116)

#### **INSTRUMENTATION:**

Automated flow injection atomic absorption spectrophotometer (AAS) system.

## REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

#### CALIBRATION:

Refer to Calcium and Magnesium tests at WAAS

### CONTROLS:

Refer to Calcium and Magnesium tests at WAAS

## *** IRON, ACID AMMONIUM OXALATE EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 1986

LIS Test Name Code

: FEEOX : DOMETOX Units

: % by wt as Fe

Work Station Code Method Code

: DOMETO2

Unit Code Supervisor : 070826 : A. Neary

Sample Type/Matrix

: Soil

## SAMPLING:

Quantity Required

: 1 g

Container

: Glass or plastic

#### SAMPLE PREPARATION:

Samples are air-dried, disaggregated and sieved to less than 2mm. A subsample is ground to <500 um (35 mesh).

### ANALYTICAL PROCEDURE:

Samples are weighed into disposable tubes. 10 mL of acid ammonium oxalate extractant is added and the tubes are capped and shaken for 4 hours in the dark. Samples are then centrifuged and the analysis is performed on the supernatant.

#### INSTRUMENTATION:

Varian AA 1275

#### REPORTING:

Maximum Significant Figures: 2

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three long term soil samples representing different soil types, 2 method blanks, 2 QC

solutions at 25% and 75% of scale, round robin ECSS samples.

Drift

:BL plus 1 standard (100% F.S.) every 10 samples.

## IRON, ACID AMMONIUM OXALATE EXTRACTABLE

## QUALITY CONTROL DATA FROM 27/01/90 TO 03/11/90

Lab: Dorset Soils

Analytical Range: - to 2.00 % by wt. Fe

## CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	**********			******	***************************************
A :	4	1.50	1.485	0.015	0.0129
B :	4	0.50	0.485	0.015	0.0208
A+B:	4	2.00	1.970	0.030	0.0182
A-B:	4	1.00	1.000	0.000	0.0294

s.d.(AB) S(between runs): 0.017

Sw(within run): 0.021 S/Sw: 0.83

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.70 - 2.30 for A+B 0.80 - 1.20 for A-B

### **RECOVERIES:**

	Number	Av. Concn	Standard(1)
	of Data	Measured	Deviation
	********		
R1:	4	0.745	0.0378
R2:	4	0.342	0.2297
R3:	4	0.217	0.1452

#### **DUPLICATES:**

Number of Data Pairs	C	Samı onçn		Mean(2) s.d.	Coefficient of var.(%)
5	0.00	•	0.20	0.011	8.6
4	0.20	:	1.00	0.012	2.0
0	1.00		2.00	N.A	N.A
9	(	)vera	1	0.012	A SAMINATORINA

	Number of Data	Data Mean	Standard(1) Deviation
			**********
Digested Blank	4	0.000	0.000

## *** IRON, CITRATE-BICARBONATE-DITHIONITE EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: FEEDI : DOMETDI Units Unit Code : % by weight Fe : 070826

Work Station Code Method Code

: 301AA5

Supervisor

: A. Neary

Sample Type/Matrix

: Soil

#### SAMPLING:

Quantity Required Container : 0.5 g dry

: Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated, and passed through a 2mm sieve. A subsample is ground and sieved to <500um. (35 mesh).

## **ANALYTICAL PROCEDURE:**

Iron is extracted from a 0.25 g soil sample using sodium citrate, sodium bicarbonate and sodium dithionite at 80°C (procedure is repeated twice). The sample is washed twice and its washings and extracts are combined and diluted to 50 mL with deionized water. The final solution is analyzed by AAS at 248.3 nm with an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale level N.B. Aluminum is determined on the same extract.

#### INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

### CONTROLS:

Calibration

:Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of

scale; 2 method blanks; round robin CSSC samples (run occasionally).

Drift

:BL plus 1 standard (100% F.S.) every 10 samples

#### NOTES:

Values for recoveries are unknown - average value used.

## IRON, CITRATE-BICARBONATE-DITHIONITE EXTRACTABLE

## QUALITY CONTROL DATA FROM 05/04/90 TO 24/11/90

Lab: Dorset Soils

Analytical Range: - to 2.00 % by wt. Fe

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
					***********
A :	5	1.5	1.514	0.014	0.030
B:	5	0.5	0.488	-0.012	0.008
A+B:	5	2.0	2.002	0.002	0.036
A-B:	5	1.0	1.026	0.026	0.025

s.d.(AB) S(between runs): 0.022

Sw(within run): 0.018 S/Sw:1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.85 - 2.15 for A+B 0.90 - 1.10 for A-B

### RECOVERIES:

	Number of Data	Av. Concn Measured	Standard(1) Deviation
	******	*********	
R1:	5	1.158	0.038
R2:	5	0.694	0.044
R3:	5	0.450	0.020

### **DUPLICATES:**

Number of Data Pairs			Sampl Concn S		Mean(2) s.d.	Coefficient of var.(%)
********		*****				***************************************
4		0.00	*	0.40	0.020	8.9
7	580	0.40	₩.	1.00	0.037	41.0
3		1.00		2.00	0.071	4.0
14			Overall	in the same	0.035	

	Number of Data	Data Mean	Standard(1) Deviation
		************	
Mehtod Blank	5	0.000	0.000

### *** IRON, SODIUM PYROPHOSPHATE EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: FEEPY : DOMETALX Units

: % by weight Fe

Work Station Code Method Code

: 703AA5

Unit Code Supervisor : 070826 : A. Neary

Sample Type/Matrix

: Soil

#### SAMPLING:

Quantity Required

: 0.6 g dry

Container

: Glass

## SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to < 2mm. A subsample is ground to <500um (35 mesh).

## ANALYTICAL PROCEDURE:

A 0.300 g quantity of sample plus 30 mL of 0.1 M sodium pyrophosphate is agitated overnight in a centrifuge tube. Samples are centrifuged at 20,000 rpm for 15 minutes and the supernatant is analyzed by AAS at 248.3 nm with an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale level

Aluminum and manganese may be determined on the same extract.

#### INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of

scale; 2 method blanks; round robin ECSS samples (run occasionally).

Drift

:BL plus 1 standard (100% F.S.) every 10 samples

#### NOTES:

Values for recoveries are unknown - average value used.

## IRON, SODIUM PYROPHOSPHATE EXTRACTABLE

## QUALITY CONTROL DATA FROM 29/03/90 TO 13/11/90

Lab: Dorset Soils

Analytical Range: - to 1.00 % by wt. Fe

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	******	*******	***********		************
<b>A</b> :	4	0.75	0.760	0.010	0.008
B :	4	0.25	0.255	0.005	0.013
A+B:	4	1.00	1.015	0.015	0.013
<b>A-B</b> :	4	0.50	0.505	0.005	0.017

s.d.(AB) S(between runs): 0.011

Sw(within run): 0.012 S/Sw:0.88

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.93 - 1.07 for A+B 0.45 - 0.55 for A-B

## **RECOVERIES:**

Number of Data		Av. Concn Measured	Standard(1) Deviation
			************
R1:	4	0.617	0.033
R2:	4	0.335	0.021
R3:	4	0.177	0.012

#### **DUPLICATES:**

Number of Data Pairs	C	Samp oncn S		Mean(2) s.d.	Coefficient of var.(%)
******				the last set one up the set on the last the	**********
8	0.00		0.20	0.009	7.6
3	0.20	-	0.50	0.015	3.9
0	0.50	F#8	1.00	N.A.	N.A
11	(	Overall		0.011	

	Number of Data	Data Mean	Standard(1) Deviation
		***********	
Method Blank	4	0.000	0.000

#### *** LEAD, ACID EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code Work Station Code

: PBUT : DOHMTE Units Unit Code : ug/g as Pb : 073882

Method Code Sample Type/Matrix : 551AA1 : Soil

Supervisor : A. Neary

#### SAMPLING:

Quantity Required Container

: 1 g dry

: Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2mm. A subsample is ground to <500um (35 mesh).

#### ANALYTICAL PROCEDURE:

A 0.500 g sample plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125°C for 2 hours. The temperature is increased to 175°C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Pb by AAS at 217.0 nm using an air-acetylene flame.

Approximate absorbance: 0.1 at the full scale value.

Copper, nickel and zinc are also determined on the extract.

#### INSTRUMENTATION:

-Varian AA1275 with programmable sample changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.2

T value: 1.0

#### CALIBRATION:

BL plus 5 standards

### CONTROLS:

Calibration

: Three long term soil samples representing different soil types;

one judiciously blended sample digest run with each run; 2

method blanks.

Drift

: BL plus 1 standard (100% F.S.) every 10 samples

#### NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal. Values for recoveries are unknown - average value used.

## LEAD, ACID-EXTRACTABLE

## QUALITY CONTROL DATA FROM 30/03/90 TO 21/11/90

Lab: Dorset Soils

Analytical Range: - to 50.0 ug/g as Pb

## CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	**********	**********	************	0400000000	
A :	5	39.00	38.94	-0.06	1.858
B :	5	16.50	14.52	-1.98	1.561
A+B:	5	55.50	53.46	-2.04	2.552
A-B:	5	22.50	24.42	. 1.92	2.295

s.d.(AB) S(between runs): 1.72 Sw(within run): 1.63 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 40.5 70.5 for A+B 12.5 32.5 for A-B

## **RECOVERIES:**

Number of Data			Av. Concn Measured	Standard(1) Deviation
			**********	*********
R1:	5		11.06	2.602
R2:	5		14.90	2.049
R3:	5	wi	27.24	2.151

## **DUPLICATES:**

Number of Sample Data Pairs Concn Span				Mean(2) s.d.	Coefficient of var.(%)
*********				*********	************
4	0.00		10.00	2.171	26.7
6	10.00		25.00	3.089	17.0
5	25.00	-	50.00	1.588	3.9
15	(	Overal	1	2.432	

	Number of Data	Data Mean	Standard(1) Deviation
		***********	***********
Digested Blank	5	0.000	0.000

## *** LEAD, TOTAL ***

#### IDENTIFICATION:

Laboratory

: Dorset Method Introduced

: 01/03/86

LIS Test Name Code Work Station Code

: PBUT : DOASV : 001PP2

Units : ug/L as Pb Unit Code : 063882 Supervisor

Method Code Sample Type/Matrix

: Streams, Lakes, Precipitation

: A. Neary

## SAMPLING:

Quantity Required

: 100 mL

Container

: 500 mL, acid washed Nalgene Teflon container, bagged in a clean room

## ANALYTICAL PROCEDURE:

Samples are acidified to 0.1% using Seastar nitric acid in a clean room. Oxygen is removed by nitrogen gas and samples are analyzed using anodic stripping voltammetry on a rotating glassy carbon disk. Change in current when lead is stripped from the glassy carbon disk is proportional to concentration.

#### INSTRUMENTATION:

EG & G 384B polarographic Analyzer with a Rotel 2 Glassy Carbon Rotating disk electrode.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.3

T value: 1.5

#### CALIBRATION:

BL plus 2 standards daily

### CONTROL:

Calibration Drift

: BL plus 2 standards, e.g. QCA and EPA standard

: End of every run (approximately every 8 samples)

## LEAD, TOTAL

## QUALITY CONTROL DATA FROM 06/01/90 TO 07/12/90

Lab: Dorset

Analytical Range: - to 2.0 ug/L as Pb

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	********				*****
A :	67	0.40	0.441	0.041	0.0843
B :	67	0.24	0.201	-0.039	0.0424
A+B:	67	0.64	0.642	0.002	0.1041
A-B:	67	0.16	0.239	0.079	0.0836

s.d.(AB) S(between runs): 0.07

Sw(within run): 0.06 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

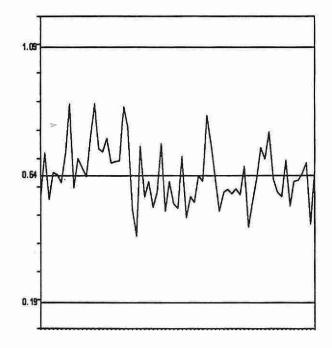
0.19 - 1.09 for A+B -0.14 - 0.46 for A-B

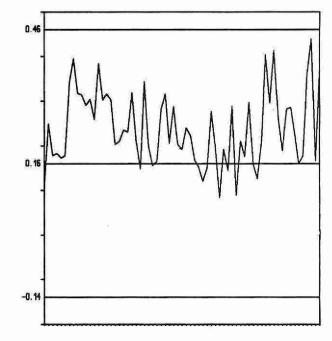
## **DUPLICATES:**

Number of Data Pairs	C	Samp onen S		Mean(2) s.d.	Coefficient of var.(%)
,				********	
9	0.0	186	0.10	0.0095	12.7
6	0.10	-	0.20	0.0291	22.6
13	0.20	9=	0.50	0.0459	14.9
6	0.50	-	2.00	0.1442	31.4
34	(	Overal	l	0.3422	

	Number of Data	Data Mean	Standard(1) Deviation
		**********	*******
Long Term Blank	67	0.0033	0.0224

## QUALITY CONTROL DATA FROM 06/01/90 TO 07/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

#### *** MAGNESIUM ***

### **IDENTIFICATION:**

Laboratory
Lis Test Name Code

: Atomic Absorption : MGUR

Method Introduced Units : 18/05/79 : mg/L as Mg

Work Station Code Method Code : PRAA400 : 001CA1 Unit Code Supervisor : 064812 : M. Young

Sample Type/Matrix

: Precipitation, Throughfall

## **SAMPLING:**

Quantity Required Container

: 5 mL : Plastic

# ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

#### **INSTRUMENTATION:**

Automated modular atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.005

T value: 0.025

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL, reslope standard every 10 samples.

## MAGNESIUM

## QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90

Lab: Atomic Absorption

Analytical Range: - to 0.500 mg/L as Mg

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			**********		
A :	78	0.30	0.2999	-0.0001	0.0022
B :	78	0.05	0.0506	0.0006	0.0011
A+B:	78	0.35	0.3504	-0.0004	0.0026
A-B:	78	0.25	0.2493	-0.0007	0.0022

s.d.(AB) S(between runs): 0.0017 Sw(within run): 0.0016 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.325 0.235 0.375 for 0.265 for A-B

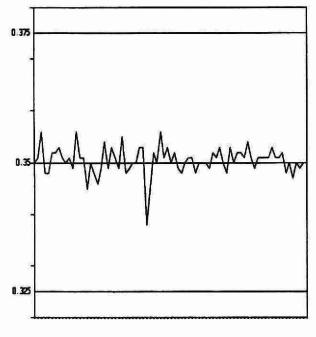
## **DUPLICATES:**

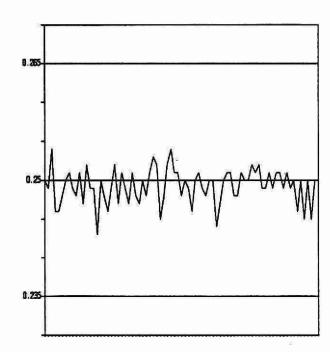
Number of Data Pairs			Mean(2) s.d.	Coefficient of var.(%)	
151	0.00		0.05	0.0008	9.4
35	0.05	1.	0.10	0.0017	2.3
17	0.10	-	0.20	0.0035	2.3
10	0.20	A Section 1	0.50	0.0054	1.4
213	C	<b>Overal</b>	l	0.0012	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
		X	
Long Term Blank	78	0.00002	0.0005

## MAGNESIUM (mg/L as Mg)

## QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

## *** MAGNESIUM ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

: 2

LIS Test Name Code

: MGUR

Method Introduced
Units

: 20/07/88 : mg/L as Mg

Work Station Code Method Code : PRAAS : 001CA1

Unit Code Supervisor : 064812 : M. Young

Sample Type/Matrix

: Rivers, Lakes

#### SAMPLING:

Quantity Required

: 5 mL

Container

: Plastic

## ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

### **INSTRUMENTATION:**

Automated modular atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.005

T value: 0.025

### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g., QCA

Drift

: BL, reslope standard every 10 samples.

### **MAGNESIUM**

## QUALITY CONTROL DATA FROM 04/01/90 TO 18/12/90

Lab: Atomic Absorption

Analytical Range: - to 2.0 mg/L as Mg

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		********			***********
<b>A</b> :	98	1.6	1.607	0.007	0.0107
<b>B</b> :	98	0.4	0.404	0.004	0.0039
A+B:	98	2.0	2.011	0.011	0.0126
A-B:	98	1.2	1.202	0.002	0.0100
<b>C</b> :	98	0.4	0.404	0.004	0.0039
D:	98	0.1	0.105	0.005	0.0018
C+D:	98	0.5	0.509	0.009	0.0044
C-D:	98	0.3	0.299	-0.001	0.0042

s.d.(AB) S(between runs): 0.008 Sw(within run): 0.007 S/Sw: 1.1

s.d.(CD) S(between runs): 0.003 Sw(within run): 0.003 S/Sw: 1.0

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91	•	2.09	for	A+B
1.14	-	1.26	for	A-B
0.41		0.59	for	C+D
0.24		0.36	for	C-D

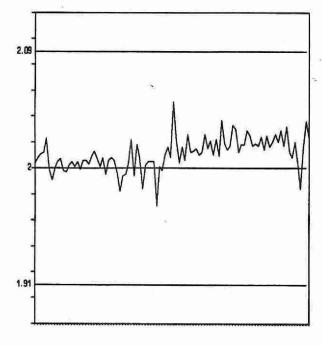
## **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
		*****		******	***********
34	0.00		0.40	0.0062	2.4
151	0.40	e-	1.00	0.0112	1.7
64	1.00	-	1.50	0.0208	1.8
22	1.50		2.00	0.0267	1.3
271	(	)veral	1	0.0143	(April 1)

	Number	Data	Standard(1)
	of Data	Mean	Deviation
	*******	(	
Long Term Blank	98	0.0001	0.0011

# MAGNESIUM (mg/L as Mg)

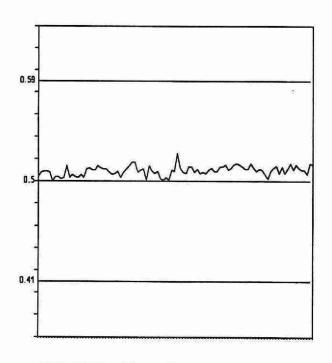
## QUALITY CONTROL DATA FROM 04/01/90 TO 18/12/90



1.2

QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B



0.36

QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

## *** MAGNESIUM ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 01/04/74

Lis Test Name Code Work Station Code : MGUR : RMAAS Units Unit Code

: mg/L as Mg : 064812

Method Code

: 0901A1

Supervisor

: M. Young

Sample Type/Matrix

: Rivers, Lakes, Soil Extracts, Stemflow.

#### SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

## ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.19 at the full scale level

#### **INSTRUMENTATION:**

Automated flow injection atomic absorption spectrophotometer (AAS) system.

## REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

#### CALIBRATION:

BL plus 11 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards e.g. QCA

Drift

: BL every 10 samples; 2 standards every 20 samples.

## **MAGNESIUM**

## QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90

Lab: Atomic Absorption

Analytical Range: - to 10.00 mg/L as Mg

## **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
		**********			************
<b>A</b> :	94	8.00	7.93	-0.07	0.0942
B :	94	2.00	1.997	-0.003	0.0306
A+B:	94	10.00	9.93	-0.07	0.1076
A-B:	94	6.00	5.94	-0.06	0.0896
C:	94	2.00	1.997	-0.003	0.0306
D:	94	0.50	0.496	-0.004	0.0191
C+D:	94	2.50	2.49	-0.01	0.0410
C-D:	94	1.50	1.5003	0.0003	0.0304

s.d.(AB) S(between runs): 0.070

Sw(within run): 0.063 S/Sw: 1.1

s.d.(CD) S(between runs): 0.025

Sw(within run): 0.021 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

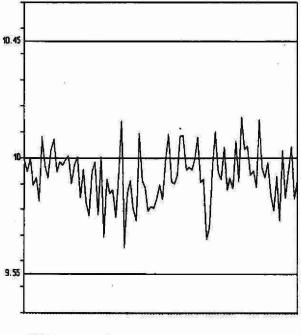
9.55	-	10.45	for	A+B
5.70	-	6.30	for	A-B
2.30	4	2.70	for	C+D
1 37	22.0	1 63	for	C-D

## **DUPLICATES:**

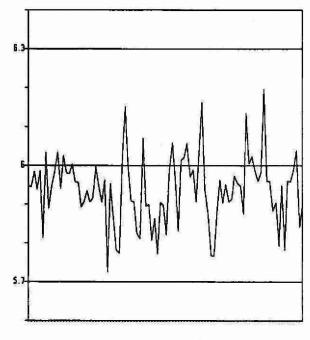
Number of Data Pairs	C	Samp oncn S		Mean(2) s.d.	Coefficient of var.(%)	
*******						
83	0.00	-	1.00	0.0167	5.1	
47	1.00	-	4.00	0.0315	1.4	
32	4.00		7.00	0.0600	1.1	
35	7.00	-	10.00	0.1014	10.1	
197	(	Overal		0.0386		

	Number of Data	Data Mean	Standard(1) Deviation
			*********
Long Term Blank	93	-0.00041	0.0151

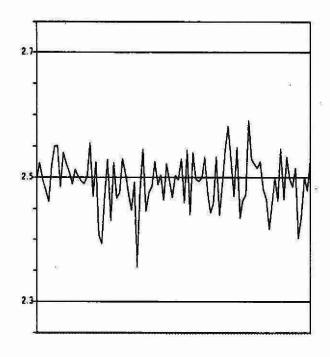
# QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90



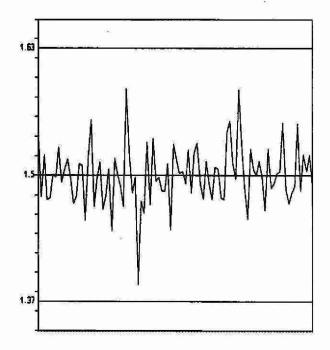
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## *** MAGNESIUM ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 08/04/86

Lis Test Name Code Work Station Code

: MGUR : WAAS

Units Unit Code

: mg/L as Mg : 064812

Method Code

: 001CA1

Sample Type/Matrix

Supervisor

: M. Young

: Domestic Waters, Leachates, Effluents, Sewage, Industrial wastes

#### SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

## ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.187 at the full scale level.

#### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.1

T value: 0.5

#### CALIBRATION:

BL plus 11 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards e.g. OCA

Drift

: BL every 10 samples; 2 standards every 20 samples

## **MAGNESIUM**

# QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90

Lab: Atomic Absorption

Analytical Range: - to 50.00 mg/L as Mg

## **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	********	***********			*******
<b>A</b> :	138	40.0	40.17	0.17	0.5417
B :	138	10.0	10.004	0.004	0.2051
A+B:	138	50.0	50.17	0.17	0.5951
A-B:	138	30.0	30.16	0.16	0.5629
<b>C</b> :	138	10.0	10.004	0.004	0.2051
D:	138	2.5	2.49	-0.01	0.0902
C+D:	138	12.5	12.498	-0.002	0.2437
C-D:	.138	7.5	7.51	0.01	0.2024

s.d.(AB) S(between runs): 0.41

Sw(within run): 0.40 S/Sw:1.03

s.d.(CD) S(between runs): 0.16

Sw(within run): 0.14 S/Sw:1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8		52.2	for	A+B
28.5	=	31.5	for	A-B
11.4		13.6	for	C+D
6.8	•	8.2	for	C-D

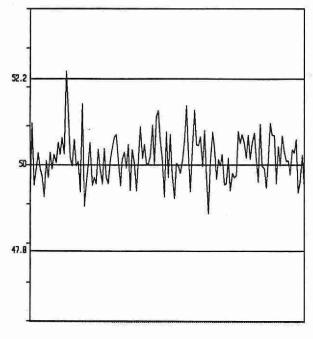
## **DUPLICATES:**

Number of Sample Data Pairs Concn Span		Mean(2) s.d.	Coefficient of var.(%)		
	****				
68	0.00		5.00	0.0597	7.2
80	5.00		10.00	0.1356	3.0
136	10.00		25.00	0.2629	5.3
68	25.00	- 1	50.00	0.4536	1.7
352	(	Overal	1	0.2302	

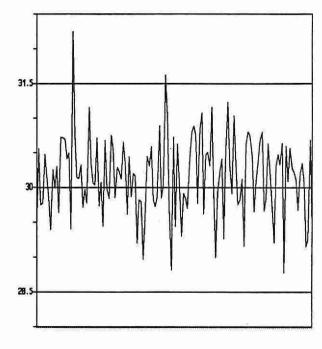
	Number	Data	Standard(1)
	of Data	Mean	Deviation
		***********	
Long Term Blank	129	-0.0452	0.1380

# MAGNESIUM (mg/L as Mg)

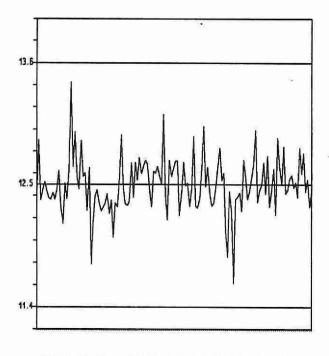
## QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90



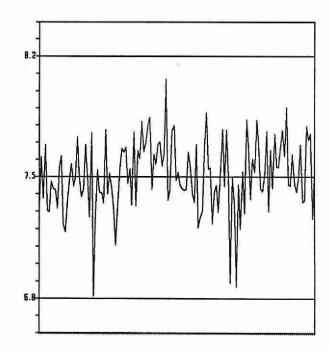
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# *** MAGNESIUM, EXCHANGEABLE CATION ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code Work Station Code : MGESC : DOCATION

Units Unit Code : meq/100 g : 355000

Method Code Sample Type/Matrix : 306AA1

Supervisor : A. Neary

## SAMPLING:

Quantity Required

: 6 g dry

: Soil

Container

: Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

#### ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for Mg by AAS at 285.2 nm with an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale level.

Aluminum, calcium, and potassium are determined on the same extract.

#### INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

## CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of

full scale; 2 method blanks; round robin ECSS samples (run occasionally).

Drift

:Bl plus 1 standard (100% Full Scale) every 10 samples.

#### NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown - average value used.

## MAGNESIUM, EXCHANGEABLE CATION

## QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90

Lab: Dorset Soils

Analytical Range: - to 2.50 meg/100 g

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	17	1.88	1.885	0.005	0.0289
B :	17	0.63	0.623	-0.007	0.0252
A+B:	17	2.50	2.509	0.009	0.0500
A-B:	17	1.25	1.262	0.012	0.0212

s.d.(AB) S(between runs): 0.027

Sw(within run): 0.015 S/Sw: 1.80

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31 - 2.69 for A+B 1.13 - 1.37 for A-B

## **RECOVERIES:**

	Number of Data	Av. Concn Measured	Standard(1) Deviation
	******		
R1:	17	0.648	0.0361
R2:	17	0.507	0.0228
R3:	17	0.180	0.0263

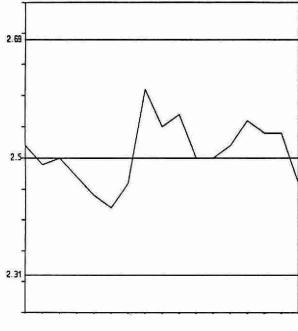
## **DUPLICATES:**

Number of Data Pairs	C	Sampl oncn S		Mean(2) s.d.	Coefficient of var.(%)
*********	****				~~~~~~~~~~
28	0.00	*	0.50	0.0240	8.4
16	0.50	<u> </u>	1.25	0.0618	7.0
7	1.25	/I <del></del>	2.50	0.0336	7.2
51	· (	Overall		0.0340	

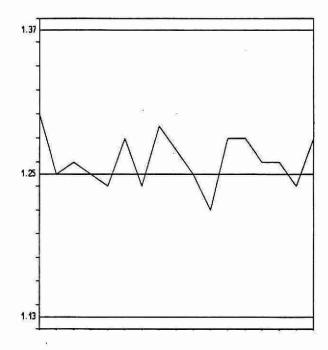
	Number of Data	Data Mean	Standard(1) Deviation
	******		
Digested Blank	17	0.000	0.000

# MAGNESIUM, EXCHANGEABLE CATION (meq/100 g as Mg)

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

## MANGANESE, ACID AMMONIUM OXALATE EXTRACTABLE

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 1986

LIS Test Name Code

: MNEOX

Units

: % by wt as Mn

Work Station Code Method Code

: DOMETOX

Unit Code Supervisor : 070825 : A. Neary

Sample Type/Matrix

: 302AA5

: Soil

## SAMPLING:

Quantity Required

Container

:Glass or plastic

## SAMPLE PREPARATION:

Samples are air-dried, disaggregated and sieved to less than 2mm. A subsample is ground to <500 um (35 mesh).

## ANALYTICAL PROCEDURE:

Samples are weighed into disposable tubes. 10 mL of acid ammonium oxalate extractant is added and the tubes are capped and shaken for 4 hours in the dark. Samples are then centrifuged and the analysis is performed on the supernatant.

#### INSTRUMENTATION:

Varian AA 1275

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.001

T value: 0.005

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three long term soil samples representing different soil samples, 2 method blanks, 2

QC solutions at 25% and 75% of scale, round robin ECSS samples.

Drift

:BL plus 1 standard (100% F.S.) every 10 samples.

## MANGANESE, ACID AMMONIUM OXALATE EXTRACTABLE

## QUALITY CONTROL DATA FROM 30/03/90 TO 14/12/90

Lab: Dorset Soils

Analytical Range: - to 0.1% by wt. Mn

## CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*********			*******	***********
A :	4	0.075	0.0620	-0.0130	0.0260
B :	4	0.025	0.0257	0.0007	0.0005
A+B:	4	0.100	0.0877	-0.0123	0.0265
A-B:	4	0.050	0.0362	-0.0138	0.0255

s.d.(AB) S(between runs): 0.0184

Sw(within run): 0.0180 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.085 - 0.115 for A+B 0.040 - 0.060 for A-B

## **RECOVERIES:**

Number of Data		Av. Concn Measured	Standard(1) Deviation
R1:	4	0.111	0.023
R2:	4	0.012	0.008
R3:	4	0.004	0.003

#### **DUPLICATES:**

Number of Sample			Mean(2)	Coefficient	
Data Pairs	Cor	ncn S	pan	s.d.	of var.(%)
	******			********	
8	0.000		0.020	0.0004	5.2
0	0.020	·	0.050	N.A.	N.A.
0	0.050	•	0.100	N.A.	N.A.
8	Ov	verall		0.0004	el .

	Number	Data	Standard(1)
	of Data	Mean	Deviation
Method Blank	4	0.000	0.000

# *** MANGANESE, CITRATE-BICARBONATE-DITHIONITE EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: MNEDI

Units

: % by weight Mn

Work Station Code

: DOMETDI

Unit Code

: 070825

Method Code Sample Type/Matrix : 301AA5 : Soil Supervisor

: A. Neary

## SAMPLING:

Quantity Required

0.5 g dry

Container : Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2mm and a subsample ground to <500um (35 mesh)

## ANALYTICAL PROCEDURE:

Manganese is extracted from a 0.25 g soil sample using sodium citrate, sodium bicarbonate and sodium dithionite at 80°C (procedure is repeated twice). The sample is washed twice and its washings and extracts are combined and diluted to 50 mL with deionized water. The final solution is analyzed by AAS at 279.5 nm with a NO₂-acetylene flame.

Approximate absorbance: 0.3 at the full scale level

N.B. Iron and Aluminum may be determined on the same extract.

#### INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.001

T value: 0.005

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three soil samples representing different soil types; two QC solutions at 25% and 75% of

full scale, 2 method blanks; round robin ECSS samples (run occasionally).

Drift

:BL plus 1 standard (100% F.S.) every 10 samples

#### NOTES:

Values for recoveries are unknown - average value used.

## MANGANESE, CITRATE-BICARBONATE-DITHIONITE EXTRACTABLE

# QUALITY CONTROL DATA FROM 05/04/90 TO 23/11/90

Lab: Dorset Soils

Analytical Range: - to 0.1 % by wt. Mn

## CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	***********		*********		*************
A :	3	0.075	0.0756	0.0006	0.0011
B :	3	0.025	0.0240	-0.0010	0.0010
A+B:	3	0.100	0.0996	-0.0004	0.0015
A-B:	3	0.050	0.0516	0.0016	0.0015

s.d.(AB) S(between runs): 0.001

Sw(within run): 0.001 S/Sw: 1.00

# **RECOVERIES:**

	Number of Data	Av. Concn Measured	Standard(1) Deviation
			V. (1997)
R1:	3	0.0997	0.0006
R2:	3	0.0127	0.0006
R3:	3	0.0037	0.0011

#### **DUPLICATES:**

Number of Data Pairs		Sample on S		Mean(2) s.d.	Coefficient of var.(%)
**********	*************			*******	
5	0.000		0.005	0.00055	12.7
3	0.005	( <del>*</del>	0.020	0.00173	11.0
0	0.020	×=	0.100	N.A	N.A
8	O	verall		0.00114	

*	Number of Data	Data Mean	Standard(1) Deviation
Method Blank	3	0.00033	0.00058

# *** MANGANESE, SODIUM PYROPHOSPHATE EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code

: MNEPY

Units Unit Code : % by weight Mn

Work Station Code Method Code : DOMETALX : 703AA5 Unit Code Supervisor : 070825 : A. Neary

Sample Type/Matrix

: 703AA : Soil

# SAMPLING:

Quantity Required

: 0.6 g dry

Container

: Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to < 2mm. A subsample is ground to <500um (35 mesh).

## ANALYTICAL PROCEDURE:

A 0.300 g quantity of sample plus 30 mL of 0.1 M sodium pyrophosphate is agitated overnight in a centrifuge tube. Samples are centrifuged at 20,000 rpm for 15 minutes and the supernatant is analyzed by AAS at 279.5 nm with an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale level

Aluminum and iron may be determined on the same extract.

#### INSTRUMENTATION:

-Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.001

T value: 0.005

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three soil samples representing different soil types; 2 QC solutions at 25% and 75% of

scale; 2 method blanks; round robin ECSS samples (run occasionally).

Drift

:BL plus 1 standard (100% F.S.) every 10 samples

#### NOTES:

Values for recoveries are unknown - average value used.

# MANGANESE, SODIUM PYROPHOSPHATE EXTRACTABLE

## QUALITY CONTROL DATA FROM 26/03/90 TO 13/11/90

Lab: Dorset Soils

Analytical Range: - to 0.05 % by wt. Mn

## CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*******			********	
<b>A</b> :	3	0.0375	0.03867	0.00117	0.00058
B :	3	0.0125	0.01333	0.00083	0.00252
A+B:	3	0.0500	0.05200	0.00200	0.00300
A-B:	3	0.0250	0.02533	0.00033	0.00208

s.d.(AB) S(between runs): 0.0018

Sw(within run): 0.0015 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.046 - 0.054 for A+B 0.023 - 0.027 for A-B

## **RECOVERIES:**

	Number of Data	08	Av. Concn Measured	Standard(1) Deviation
R1:	3		0.0607	0.00737
R2:	3		0.0043	0.00058
R3:	3		0.0013	0.00058

#### **DUPLICATES:**

Number of Data Pairs	**		Samp		Mean(2) s.d.	Coefficient of var.(%)
6		0.000		0.010	0.00029	10.6
2		0.010		0.025	0.00200	12.1
0		0.025	-	0.050	N.A	N.A
8			verall	•	0.00103	

	Number of Data	Data Mean	Standard(1) Deviation	
		***********		
Method Blank	3	0.000	0.000	

## *** NICKEL, ACID. EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code Work Station Code : NIUT : DOHMTE Units Unit Code : ug/g as Ni : 073828

Method Code

: 551AA1

Supervisor

: A. Neary

Sample Type/Matrix

: Soil

#### **SAMPLING:**

Quantity Required

: 1 g dry

Container

: Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2mm. A subsample is ground to <500um (35 mesh).

#### ANALYTICAL PROCEDURE:

A 0.500 g sample plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125°C for 2 hours. The temperature is increased to 175°C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Ni by AAS at 232.0 nm using an air-acetylene flame.

Approximate absorbance: 0.2 at the full scale value.

Copper, lead and zinc are also determined on the same extract.

#### **INSTRUMENTATION:**

- -Varian AA1275 with programmable sample changer and Gilson Minipuls II pump
- -Balance accurate to 0.001 g

## REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.2

T value: 1.0

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

: Three long term soil samples representing different soil types, 2 method blanks and one

judiciously blended sample extract run with each run.

Drift

: BL plus 1 standard (100% F.S.) every 10 samples

## NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal. Values for recoveries are unknown - average value used.

## NICKEL, ACID-EXTRACTABLE

## QUALITY CONTROL DATA FROM 30/03/90 TO 22/11/90

Lab: Dorset Soils

Analytical Range: - to 50.0 ug/g as Ni

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
					***************************************
A :	4	36.30	35.95	0.35	1.667
B :	4	13.50	12.85	0.65	2.353
A+B:	4	49.80	48.80	-1.00	3.677
A-B:	4	22.80	23.10	0.30	1.762

s.d.(AB) S(between runs): 2.04

Sw(within run): 1.25 S/Sw: 1.64

On any given day the calibration is accepted if the values obtained lie within the ranges:

42.3 - 57.3 for A+B 17.8 - 27.8 for A-B

## **RECOVERIES:**

Number of Data		Av. Concn Measured	Standard(1) Deviation
	******	******	
R1:	5	7.58	1.361
R2:	5	28.02	2.517
R3:	5	6.14	1.824

## **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
**********			••••••	********	
9	0.00	•//	10.00	1.224	22.9
6	10.00	-	25.0	1.401	7.1
0	25.00	•:	50.0	N.A	N.A
15	(	Overal	1	1.396	

	Number of Data	Data Mean	Standard(1) Deviation
Digested Blanks	5	0.48	0.455

## *** NITROGEN, AMMONIA PLUS AMMONIUM ***

#### **IDENTIFICATION:**

Laboratory

: Dorset

Method Introduced

: 01/06/76

LIS Test Name Code Work Station Code

: NNHTFR : DONUT

Units Unit Code : ug/L as N : 063807

Method Code

: 1524C2

Supervisor

Sample Type/Matrix

: Streams, Lakes, Precipitation, and Soil Leachates

: A. Neary

#### SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix

Approximate absorbance: 0.40 at the full scale level. Nitrate plus nitrite is determined simultaneously.

## INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

#### CALIBRATION:

BL plus 8 standards

#### CONTROLS:

Calibration

: LTBL plus 4 QC standards, e.g. QCA

Drift

: BL every 10 samples and BL plus check standard every 20 samples

# NITROGEN, AMMONIA PLUS AMMONIUM

# QUALITY CONTROL DATA FROM 05/01/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 1000 ug/L as N

## CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
				********	
A :	88	750.0	749.8	-1.2	6.18
B :	88	250.0	248.4	-1.6	4.91
A+B:	88	1000.0	998.1	-1.9	9.77
A-B:	88	500.0	501.4	1.4	5.41
<b>C</b> :	88	75.0	73.6	-1.4	2.15
D:	88	25.0	24.6	-0.4	1.45
C+D:	88	100.0	98.2	-1.8	3.02
C-D:	88	50.0	49.0	-1.0	2.07

s.d.(AB) S(between runs): 5.6

Sw(within run): 3.8 S/Sw: 1.50

s.d.(AB) S(between runs): 1.8

Sw(within run): 1.5 S/Sw: 1.25

On any given day the calibration is accepted if the values obtained lie within the ranges:

970		•	1030	for	A+B
480	2		520	for	A-B
88		•	112	for	C+D
42		-	58	for	C-D

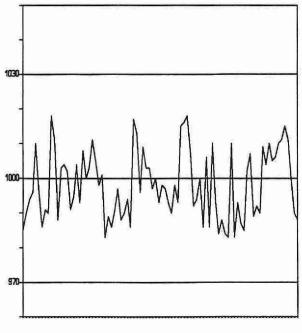
## **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
174	0.0	-	25.0	1.30	19.1
40	25.0	Upp.	50.0	1.75	3.8
28	50.0	129	500.0	3.80	1.5
4	500.0	18	1000.0	4.42	0.5
246		Overal		1.63	0.0

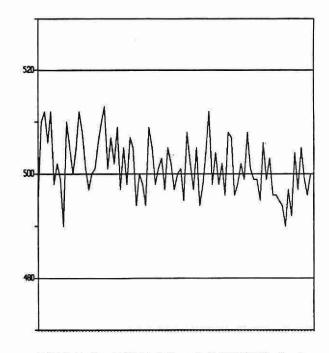
	Number of Data	Data Mean	Standard(1) Deviation
	************		****
Long Term Blank Reference Check	88 79	0.863 197.1	1.297 5.590

# NITROGEN, AMMONIA PLUS AMMONIUM (ug/L as N)

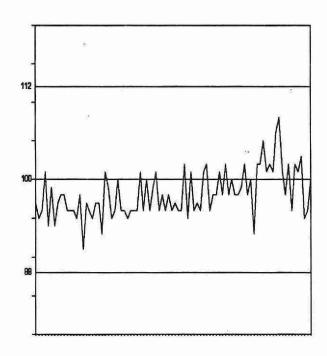
QUALITY CONTROL DATA FROM 05/01/90 TO 21/12/90



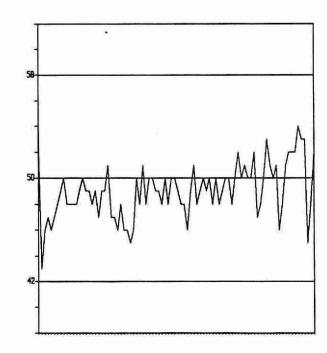
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## NITROGEN, AMMONIA PLUS AMMONIUM ***

## **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code

Method Code Sample Type/Matrix : Colourimetry : NNHTFR

Method Introduced

Units Unit Code Supervisor

: ug/fltr as N : 361807 : M. Rawlings

: 01/05/84

: Dry deposition air filter extracts

## SAMPLING:

Quantity Required

: 10 mL

: PRAM

: 004AI1

Container

: 50 mL Polyethylene tube

#### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on an extract from a dry deposition air filter via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects. Ammonia plus ammonium for precipitation, throughfall, and stemflow samples is also determined at this work station.

Approximate absorbance: 0.7 at the full scale level.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

#### CALIBRATION:

BL plus 7 standards

## CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples, standard every 20 samples.

# NITROGEN, AMMONIA PLUS AMMONIUM

## QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90

Lab: Colourimetry

Analytical Range: - to 50.0 ug/filter as N

## **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
					************
A :	128	40	40.25	0.25	0.245
B :	128	20	20.06	0.06	0.158
A+B:	128	60	60.31	0.31	0.317
A-B:	128	20	20.19	0.19	0.265
C :	128	20	20.06	0.06	0.158
D:	128	4	4.10	0.10	0.134
C+D:	128	24	24.16	0.16	0.240
C-D:	128	16	15.96	-0.04	0.169

s.d.(AB) S(between runs): 0.21

Sw(within run): 0.19 S/Sw: 1.10

s.d.(CD) S(between runs): 0.15

Sw(within run): 0.12 S/Sw: 1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

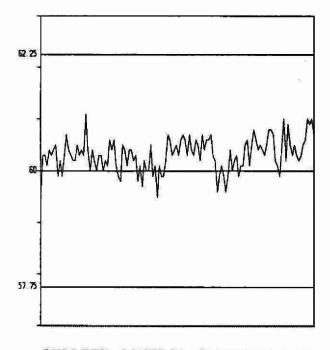
57.75	-	62.25	for	A+B
18.5	<b>—</b>	21.5	for	A-B
23.0	-	25.0	for	C+D
15.4	-	16.6	for	C-D

## **DUPLICATES:**

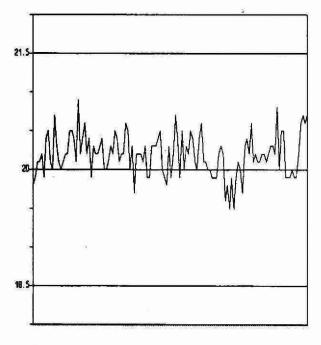
Number of	Sample			of Sample Mean(2)		Coefficient
Data Pairs	Concn Span		pan	s.d.	of var.(%)	
	*****		*****			
92	0.00	•	5.00	0.084	0.6	
65	5.00		10.00	0.099	1.4	
113	10.00		50.00	0.132	1.3	
270	(	Overall		0.103		

	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	128	-0.146	0.435

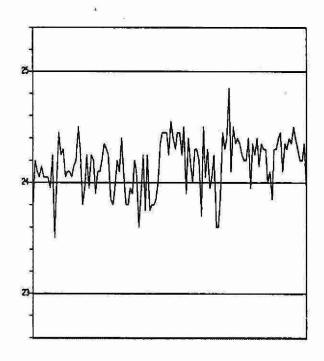
QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90



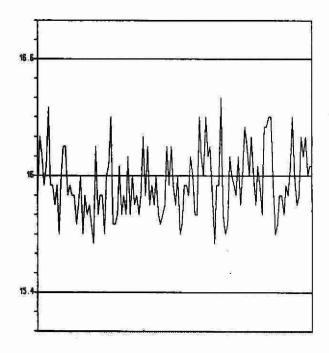
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## *** NITROGEN, AMMONIA PLUS AMMONIUM ***

#### **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: 01/05/84

LIS Test Name Code

: NNHTFR, NNHTUR : PRAM Units Unit Code : mg/L as N : 064807

Work Station Code Method Code

: 103CC3, 003CC3

Supervisor

: M. Rawlings

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

#### SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

## **ANALYTICAL PROCEDURE:**

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects. Ammonia plus ammonium for dry deposition air filter extracts is also determined at this work station.

Approximate absorbance: 0.7 at the full scale level.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5cm light path at 630 nm. Data capture, reduction and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.002

T value: 0.01

## CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples, standard every 20 samples

# NITROGEN, AMMONIA PLUS AMMONIUM (NNHTFR)

## QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90

Lab: Colourimetry

Analytical Range: - to 2.0 mg/L as N

## **CALIBRATION CONTROL:**

9	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
			*********	***	
A :	128	1.60	1.609	0.009	0.0106
B :	128	0.80	0.801	0.001	0.0064
A+B:	128	2.40	2.411	0.011	0.0134
A-B:	128	0.80	0.808	0.008	0.0112
<b>C</b> :	128	0.80	0.801	0.001	0.0064
D:	128	0.16	0.164	0.004	0.0054
C+D:	128	0.96	0.965	0.005	0.0097
C-D:	128	0.64	0.637	-0.003	0.0067

s.d.(AB) S(between runs): 0.0087 Sw

Sw(within run): 0.0079 S/Sw: 1.1

s.d.(CD) S(between runs): 0.0059

Sw(within run): 0.0047 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31 - 2.49 for A+B 0.74 - 0.86 for A-B 0.92 - 1.00 for C+D 0.616 - 0.664 for C-D

## **DUPLICATES:**

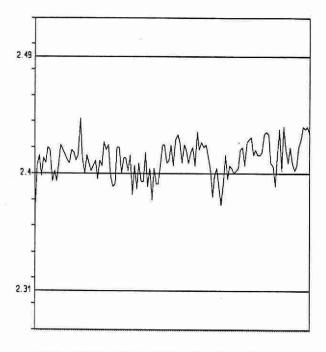
Number of Data Pairs	Sample Conen Span		Mean(2) s.d.	Coefficient of var.(%)	
119	0.00	A.	0.20	0.0031	11.0
166	0.20	-	1.00	0.0039	1.0
30	1.00	,	1.50	0.0061	0.5
10	1.50	-	2.00	0.0089	0.6
325	(	Overall		0.0038	200

	Number of Data	Data Mean	Standard(1) Deviation
	***************************************		
Long Term Blank	128	-0.0058	0.0174

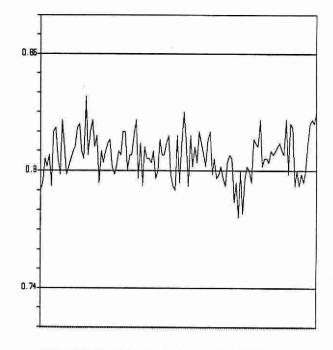
# NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)

(NNHTFR)

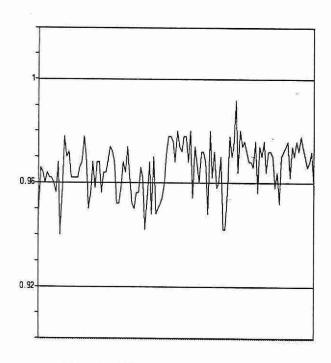
QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90



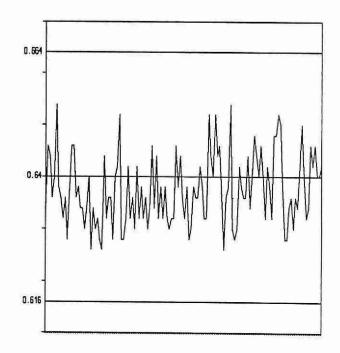
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# NITROGEN, AMMONIA PLUS AMMONIUM (NNHTUR)

# QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90

Lab: Colourimetry

Analytical Range: - to 2.0 mg/L as N

## **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			***********		
A :	128	1.6	1.609	0.009	0.0106
B :	128	0.8	0.801	0.001	0.0064
A+B:	128	2.4	2.410	0.010	0.0134
A-B:	128	0.8	0.808	0.008	0.0112
C :	128	0.8	0.801	0.001	0.0064
D:	128	0.16	0.164	0.004	0.0054
C+D:	128	0.96	0.965	0.005	0.0097
C-D:	128	0.64	0.637	-0.003	0.0067

s.d.(AB) S(between runs): 0.009

Sw(within run): 0.008 S/Sw: 1.10

s.d.(CD) S(between runs): 0.006

Sw(within run): 0.005 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31 2.49 for A+B 0.74 0.86 for A-B 0.92 1.00 for C+D 0.616 0.664 for C-D

## **DUPLICATES:**

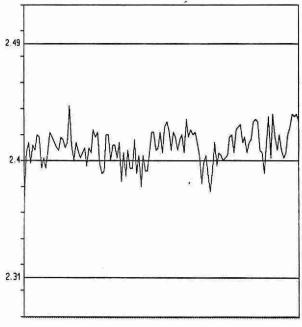
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
				******	**********
119	0.00	ė.	0.20	0.0027	10.8
168	0.20		1.00	0.0038	1.0
40	1.00	-	2.00	0.0076	0.6
327	(	Overall		0.0037	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
	********	•••••	
Long Term Blank	128	-0.0046	0.0076

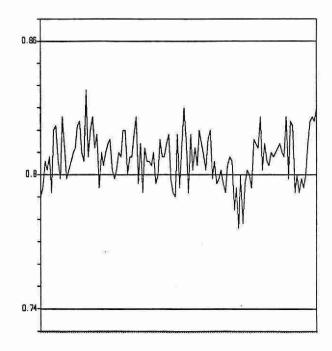
## NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N

(NNHTUR)

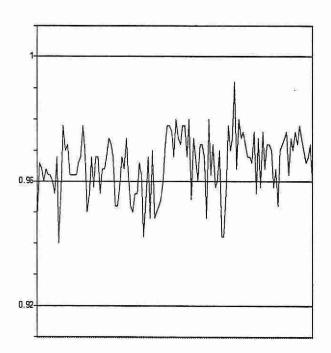
QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90



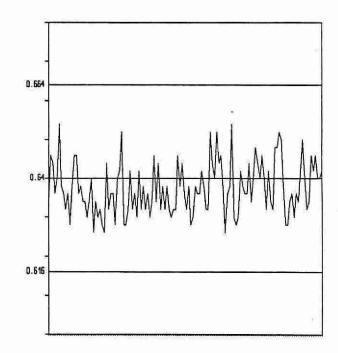
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## *** NITROGEN, AMMONIA PLUS AMMONIUM ***

## **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code Method Code

Sample Type/Matrix

: Colourimetry : NNHTFR

: RNDNP

: 103DC2

Method Introduced Units

: 01/04/78 : mg/L as N : 064807

Unit Code

Supervisor : Rivers, Lakes, Soil Extracts, Effluents

: M. Rawlings

#### SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.5 at the full scale level.

Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.002

T value: 0.01

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

## NITROGEN, AMMONIA PLUS AMMONIUM

## QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 2.0 mg/L as N

## **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias .	Deviation
A :	118	1.6	1.5996	-0.0004	0.0091
B :	118	0.8	0.8003	0.0003	0.0079
A+B:	118	2.4	2.4000	0.0000	0.0122
A-B:	118	0.8	0.7993	-0.0007	0.0118
C :	118	0.8	0.8003	0.0003	0.0079
D:	118	0.16	0.1592	-0.0008	0.0006
C+D:	118	0.96	0.9596	-0.0004	0.0120
C-D:	118	0.64	0.6412	0.0012	0.0059

s.d.(AB) S(between runs): 0.0085

Sw(within run): 0.0083 S/Sw: 1.02

s.d.(CD) S(between runs): 0.007

Sw(within run): 0.004 S/Sw: 1.68

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31 - 2.49 for A+B 0.74 - 0.86 for A-B 0.92 - 1.0 for C+D 0.616 - 0.664 for C-D

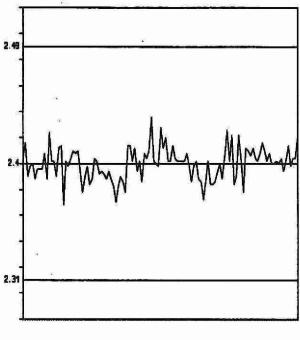
## **DUPLICATES:**

Number of Data Pairs	irs Concn Span		pan	Mean(2) s.d.	Coefficient of var.(%)
				,	
110	0.00	-	0.02	0.003	38.1
122	0.02	-	0.20	0.006	14.6
12	0.20	-	1.00	0.015	4.0
0	1.00	-	2.00	N.A	N.A
244	(	Overall		0.005	one socialismo

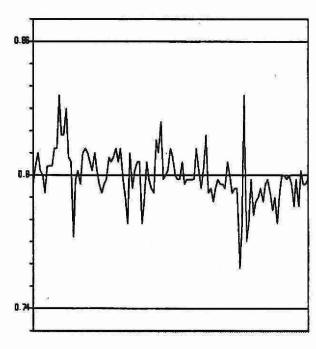
	Number of Data	Data Mean	Standard(1) Deviation
100			***********
Long Term Blank	117	0.0018	0.0054

# NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)

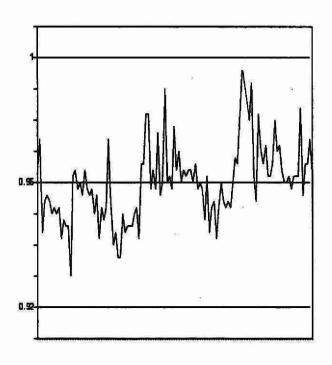
QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90



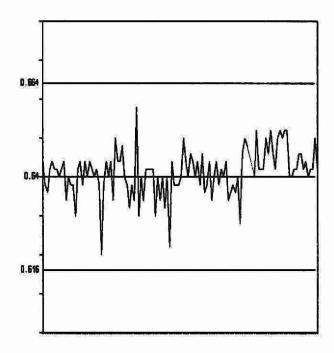
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

## *** NITROGEN, AMMONIA PLUS AMMONIUM ***

#### **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: 01/04/77

LIS Test Name Code Work Station Code : NNHTFR : SDNP Units Unit Code : mg/L as N : 064807

Method Code

: 103AC2

Supervisor

: M. Rawlings

Sample Type/Matrix

: Sewage, Industrial Waste, Leachate, Domestic Waters, Effluents

#### SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

## **ANALYTICAL PROCEDURE:**

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.7 at the full scale level.

Reactive orthophosphate, nitrogen-nitrite and nitrogen-nitrate plus nitrite are determined simultaneously.

#### **INSTRUMENTATION:**

Basic automated modular continuous flow system plus one 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture, reduction, and processing via a multistage microcomputer system.

## REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. OCA

Drift

: BL every 10 samples; standard every 20 samples

## NITROGEN, AMMONIA PLUS AMMONIUM

## QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90

Lab: Colourimetry

Analytical Range: - to 50.0 mg/L as N

## **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	*********		55 C5500000000	*********	***************************************
A :	153	40.0	40.086	0.086	0.267
B :	153	20.0	20.061	0.061	0.169
A+B:	153	60.0	60.146	0.146	0.320
A-B:	153	20.0	20.025	0.025	0.311
<b>C</b> :	153	20.0	20.061	0.061	0.169
D:	153	4.0	4.006	0.006	0.068
C+D:	153	24.0	24.067	0.067	0.197
C-D:	153	16.0	16.054	0.054	0.166

s.d.(AB) S(between runs): 0.223

Sw(within run): 0.220 S/Sw: 1.01

s.d.(CD) S(between runs): 0.129

Sw(within run): 0.117 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

57.75	•	62.25	for	A+B
18.5		21.5	for	A-B
23.1		24.9	for	C+D
15.4		16.6	for	C-D

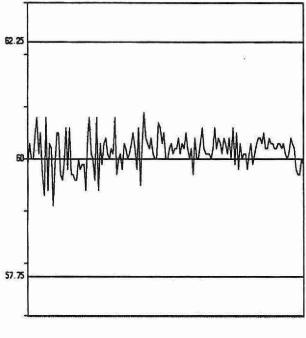
## **DUPLICATES:**

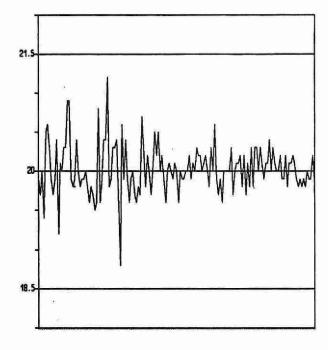
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
***************				*********	***************************************
136	0.00	<b>(4)</b>	0.20	0.0332	79.3
73	0.20	±2×	2.00	0.0528	24.2
58	2.00	•	10.00	0.1344	5.6
37	10.00	-	20.00	0.2325	1.7
10	20.00	<b>4</b> 1	50.00	0.5712	4.9
314	(	Overal	1	0.0782	

	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	153	-0.00098	0.023

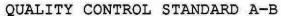
# NITROGEN, AMMONIA PLUS AMMONIUM (mg/L as N)

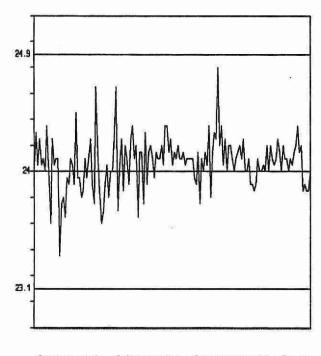
QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90





QUALITY CONTROL STANDARD A+B





QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

____ CONTROL LIMIT

# *** NITROGEN, NITRATE ***

#### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/78

LIS Test Name Code
Work Station Code

: NNO3UR : PRIC1

Units
Unit Code

: mg/L as N

Method Code

: 003AI0

Supervisor

: 064807 : F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

#### SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

## **ANALYTICAL PROCEDURE:**

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards.

Sulphate and chloride are determined simultaneously.

#### **INSTRUMENTATION:**

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

#### NOTES:

Two analytical ranges are in operation at this work station, and subsequently, quality control results are provided for each range.

## NITROGEN, NITRATE

# QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90

Lab: Ion Chromatography

Analytical Range: - to 1 mg/L as N

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	44	Standard(1) Deviation
				*********		
A :	45	0.8	0.7999	-0.0001		0.0162
B :	45	0.2	0.1899	-0.0101		0.0180
A+B:	45	1.0	0.9897	-0.0103		0.0306
A-B:	45	0.6	0.6100	0.0100		0.0153

s.d.(AB) S(between runs): 0.017 Sw(within run): 0.011 S/Sw: 1.58

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.95 - 1.05 for A+B 0.57 - 0.63 for A-B

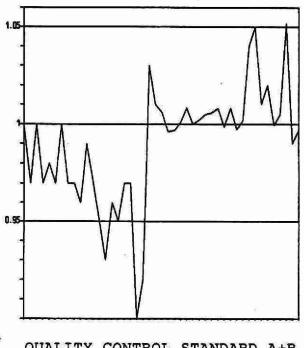
## **DUPLICATES:**

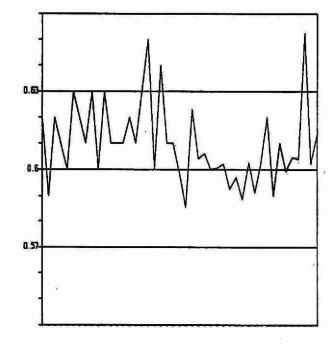
Number of Data Pairs	Sample Concn Span		• Mean(2) . s.d.	Coefficient of var.(%)	
				*******	
23	0.00	-	0.20	0.0049	4.0
44	0.20	-	0.50	0.0072	2.0
50	0.50	-	1.00	0.0107	2.1
117	C	veral	1	0.0080	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
		***********	
Long Term Blank	45	0.006	0.0121

# NITROGEN, NITRATE (mg/L as N)

## QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

# NITROGEN, NITRATE

# QUALITY CONTROL DATA FROM 08/01/90 TO 28/12/90

Lab: Ion Chromatography

Analytical Range: - to 2 mg/L as N

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
					*
A :	36	1.6	1.605	0.005	0.0240
B :	36	0.4	0.408	0.008	0.0128
A+B:	36	2.0	2.014	0.014	0.0343
A-B:	36	1.2	1.197	-0.003	0.0172

s.d.(AB) S(between runs): 0.019 Sw(within run): 0.012 S/Sw: 1.58

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 - 2.09 for A+B 1.14 - 1.26 for A-B

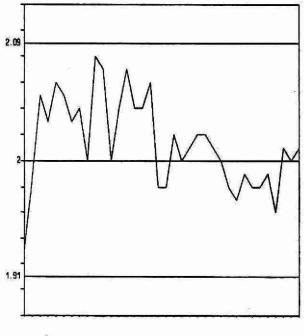
### **DUPLICATES:**

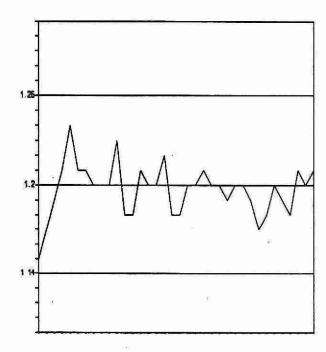
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
********					
19	0.00		0.02	0.0043	30.8
48	0.02	•	0.20	0.0069	6.9
28	0.20	•	2.00	0.0076	1.2
95	C	)veral	l common	0.0067	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
			**********
Long Term Blank	36	0.0125	0.0264

NITROGEN, NITRATE (mg/L as N)

QUALITY CONTROL DATA FROM 08/01/90 TO 28/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

### *** NITROGEN, NITRATE ***

### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/07/80

LIS Test Name Code

: NNO3UR

Units

: ug/Filter as N

Work Station Code Method Code : PRLOV : 004AIC Unit Code Supervisor : 361807 : F. Lo

Sample Type/Matrix

: W40 filters from LoVol filter packs

### SAMPLING:

Quantity Required

: 1 filter

Container

: 50 mL polypropylene tube

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards. Results are converted to ug/filter as N.

Chloride and sulphate are determined simultaneously.

### **INSTRUMENTATION:**

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

#### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

# NITROGEN, NITRATE

# QUALITY CONTROL DATA FROM 19/01/90 TO 20/12/90

Lab: Ion Chromatography

Analytical Range: - to 100 µg/filter as N

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*********		***********		
A :	24	80.0	79.3	-0.7	0.82
B :	24	20.0	19.9	-0.1	0.49
A+B:	24	100.0	99.2	-0.8	1.01
A-B:	24	60.0	59.5	-0.5	0.90

s.d.(AB) S(between runs): 0.67

Sw(within run): 0.63

S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

95.5 - 104.5 57.0 - 63.0

for A+B for A-B

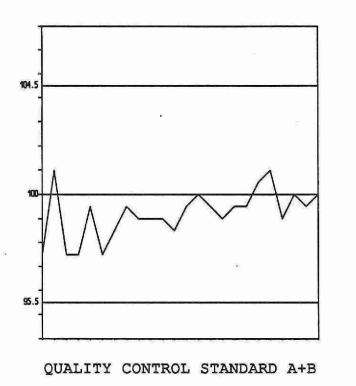
### **DUPLICATES:**

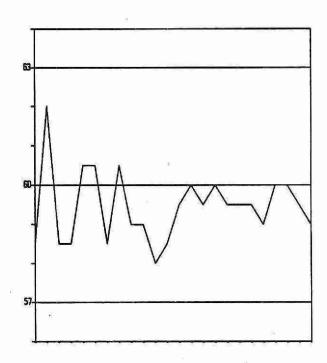
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
		****			
16	0.0	<b>*</b>	25.0	0.31	3.6
5	25.0	-	50.0	0.39	1.3
8	50.0		100.0	0.63	0.9
29	(	Overal	1	0.46	W.

	Number	Data	Standard(1)
	of Data	Mean	Deviation
	8	***	
Long Term Blank	24	0.167	0.381

NITROGEN, NITRATE (ug/filter as N)

QUALITY CONTROL DATA FROM 19/01/90 TO 20/12/90





QUALITY CONTROL STANDARD A-B

### *** NITROGEN, NITRATE ***

### **DENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/07/80

LIS Test Name Code

: NNO3FR,NNRICF

Units

: ug/Filter as N

Work Station Code Method Code

: PRSEO : 004AI0 Unit Code Supervisor

: 361807 : F. Lo

Sample Type/Matrix

: Nylon (NNRICF) filter from LoVol and sequential filter packs, and Teflon

(NN03FR) filters from sequential filter packs.

#### SAMPLING:

**Quantity Required** 

: 1 filter

Container

: 50 mL polypropylene tube

#### SAMPLE PREPARATION:

Filters are extracted with 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03 N NaOH (nylon) in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₂/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by the comparison of the sample peak heights to a series of standards. Results are converted to ug/filter as N. Chloride and sulphate are determined simultaneously.

### INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1.0

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. OCA

Drift

: 1 standard every 10 samples

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

## NITROGEN, NITRATE (NNO3FR)

# QUALITY CONTROL DATA FROM 03/01/90 TO 14/12/90

Lab: Ion Chromatography

Analytical Range: - to 50 ug/filter as N

### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*****	*******			
A :	94	40.0	39.7	-0.3	0.472
B :	94	10.0	9.9.	-0.1	0.278
A+B:	94	50.0	49.6	-0.4	0.542
A-B:	94	30.0	29.7	-0.3	0.553

s.d.(AB) S(between runs): 0.39 Sw(within run): 0.39 S/Sw: 1.0

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 - 52.2 for A+B 28.5 - 31.5 for A-B

### **DUPLICATES:**

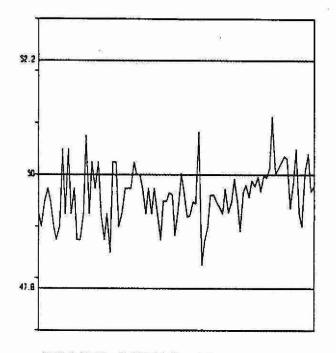
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
			*********	5000000000000000	
158	0.0	-	5.0	0.157	9.3
86	5.0		25.0	0.226	2.2
16	25.0	-	50.0	0.391	1.3
260	(	Overal	I	0.189	1.4

	Number of Data	Data Mean	Standard(1) Deviation
			********
Long Term Blank	94	0.0691	0.195

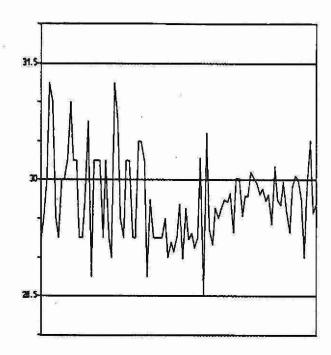
# NITROGEN, NITRATE (ug/filter as N)

(NNO3FR)

QUALITY CONTROL DATA FROM 03/01/90 TO 14/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

### NITROGEN, NITRATE (NNRICF)

# QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Ion Chromatography

Analytical Range: - to 50 ug/filter as N

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		*********	*******	********	
A :	87	40.0	39.81	-0.19	0.462
B :	87	10.0	9.97	-0.03	0.286
A+B:	87	50.0	49.78	-0.22	0.602
A-B:	87	30.0	29.83	-0.17	0.477

s.d.(AB) S(between runs): 0.38 Sw(within run): 0.34 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 52.2 for A+B 28.5 31.5 for A-B

### **DUPLICATES:**

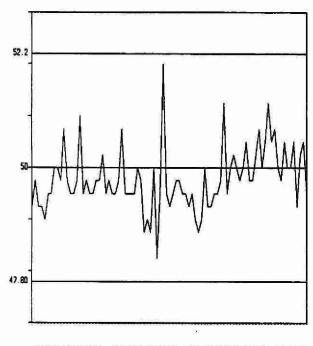
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
35	0.0	-	1.5	0.122	14.9
57	1.5	•	5.0	0.183	6.1
75	5.0	-	50.0	0.212	2.0
167	3	Overal	l	0.181	

P41	Number	Data	Standard(1)
	of Data	Mean	Deviation
	(	******	
Long Term Blank	87	0.109	0.295

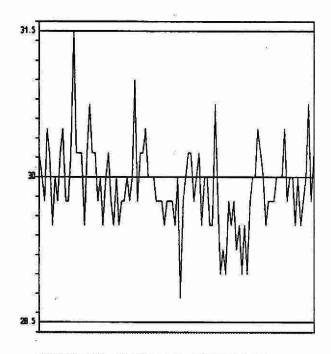
# NITROGEN, NITRATE (ug/filter as N)

(NNRICF)

QUALITY CONTROL DATA FROM 03/1/90 TO 20/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

### *** NITROGEN, NITRATE PLUS NITRITE ***

### **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code Method Code Sample Type/Matrix : Dorset : NNOTFR

: DONUT

Method Introduced Units

Unit Code

: 13/06/78 : ug/L as N : 063807 : A. Neary

: 1525C2 Supervisor : Streams, Lakes, Precipitation, and Soil Leachates

#### SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a sample. Nitrate is reduced to nitrite in alkaline media at 37°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.4 at the full scale level.

Ammonia plus ammonium is determined simultaneously.

#### **INSTRUMENTATION:**

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 2

T value: 10

### CALIBRATION:

BL plus 8 standards

#### CONTROLS:

Calibration:

LTBL plus 4 QC standards, e.g. QCA

Drift:

BL every 10 samples and BL plus check standard every 20 samples.

# NITROGEN, NITRATE PLUS NITRITE

# QUALITY CONTROL DATA FROM 07/02/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 1000 ug/L as N

## **CALIBRATION CONTROL:**

s	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*********		*********		
<b>A</b> :	79	750	749.5	-0.5	6.05
B :	79	250	249.0	-1.0	3.68
A+B:	79	1000	998.5	-1.5	8.74
A-B:	79	500	500.4	0.4	4.91
<b>C</b> :	79	75	73.9	-1.1	2.11
D:	79	25	24.4	-0.6	1.25
C+D:	79	100	98.3	-1.7	3.00
C-D:	79	50	49.5	-0.5	1.74

s.d.(AB) S(between runs): 3.0

Sw(within run): 3.5 S/Sw: 0.86

On any given day the calibration is accepted if the values obtained lie within the ranges:

970 - 1030 for A+B 480 - 520 for A-B 88 - 112 for C+D 42 - 58 for C-D

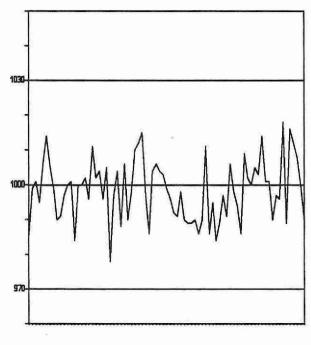
## **DUPLICATES:**

Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
*******			*********	*******	
88	0.0		25.0	1.31	15.2
28	25.0		50.0	1.78	4.1
34	50.0	-	100.0	3.57	4.3
61	100.0		500.0	6.97	3.6
8	500.0		1000.0	24.07	3.3
219	9 (20 13)	Overa	11	3.06	<u>~</u>

	Number	Data	Standard(1)
	of Data	Mean	Deviation
		~~~	************
Long Term Blank	79	0.67	1.288

NITROGEN, NITRATE PLUS NITRITE (ug/L as N)

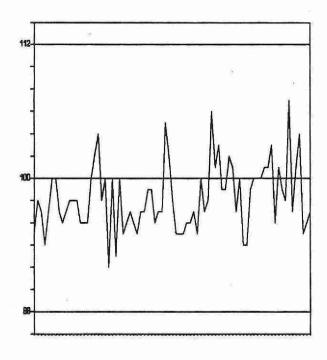
QUALITY CONTROL DATA FROM 07/02/90 TO 21/12/90

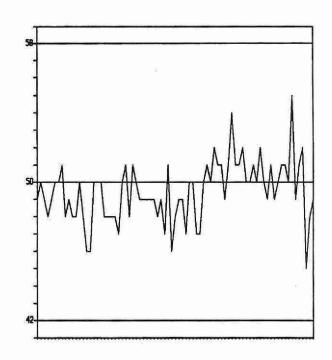


500 500 480

QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B





QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

*** NITROGEN, NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code Method Code

Sample Type/Matrix

: Colourimetry : NNOTFR

: RNDNP

: 102DC2

Method Introduced

Units Unit Code

Supervisor

: 064807 : M. Rawlings

: 01/04/78

: mg/L as N

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents

SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.005

T value: 0.025

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. OCA

Drift

: BL every 10 samples; standard every 20 samples

Interference

: Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective interference suppression.

Recovery

: Individual nitrate and nitrite standards of equal N concentration show effectiveness of

reduction step.

NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 5.0 mg/L as N

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	****			******	***********
A :	123	4.0	4.0003	0.0003	0.031
B :	123	2.0	2.0037	0.0037	0.020
A+B:	123	6.0	6.0041	0.0041	0.043
A-B:	123	2.0	1.9966	-0.0034	0.029
C :	123	2.0	2.0037	0.0037	0.020
D:	123	0.4	0.4018	0.0018	0.009
C+D:	123	2.4	2,4056	0.0056	0.025
C-D:	123	1.6	1.6019	0.0019	0.018

s.d.(AB) S(between runs): 0.03 Sw(v

Sw(within run): 0.02 S/Sw: 1.3

s.d.(CD) S(between runs): 0.02

Sw(within run): 0.01 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

5.77		6.23	for	A+B
1.85	-	2.15	for	A-B
2.30		2.50	for	C+D
1.54		1.66	for	C-D

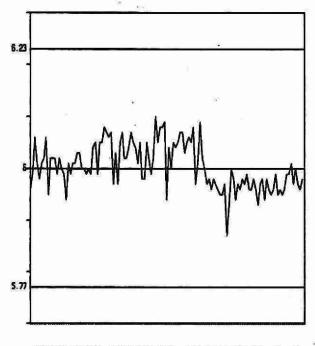
DUPLICATES:

Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
				**********	**************
111	0.00	-	0.20	0.0087	17.1
165	0.20	•	1.00	0.0101	4.5
30	1.00		2.50	0.0294	2.5
0	2.50	-	5.00	N.A.	N.A.
306	Overall		1	0.0107	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
	********	*********	***********
Long Term Blank	123	0.003	0.008

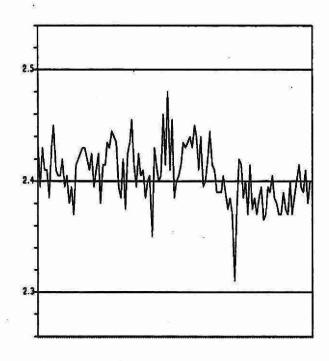
NITROGEN, NITRATE PLUS NITRITE (mg/L as N)

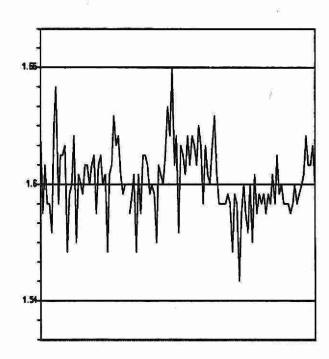
QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90



QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B





QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

*** NITROGEN, NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory

LIS Test Name Code Work Station Code

Method Code Sample Type/Matrix : Colourimetry : NNOTFR

Method Introduced Units

Unit Code Supervisor

: 064807 : M. Rawlings

: 01/04/78

: mg/L as N

: Sewage, Industrial Waste, Leachate, Domestic Waters

SAMPLING:

Ouantity Required

: 10 mL

: SDNP : 102CC2

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.7 at the full scale level.

Ammonia plus ammonium, nitrite, and reactive phosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 38°C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter. Data capture, reduction, and processing via a multi - stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

Interference

: Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms

effective interference suppression.

Recovery

: Individual nitrate and nitrite standards of equal N concentration show effectiveness of

reduction step.

NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90

Lab: Colourimetry

Analytical Range: - to 50.0 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	149	40.0	40.008	0.008	0.346
B :	149	20.0	20.086	0.086	0.188
A+B:	149	60.0	60.094	0.0094	0.444
A-B:	149	20.0	19.923	-0.077	0.336
C :	149	20.0	20.086	0.086	0.188
D:	149	4.0	4.018	0.018	0.067
C+D:	149	24.0	24.104	0.104	0.218
C-D:	149	16.0	16.068	0.068	0.179

s.d.(AB) S(between runs): 0.278

Sw(within run): 0.238 S/Sw: 1.17

s.d.(CD) S(between runs): 0.141

Sw(within run): 0.127 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

61.8 21.2 58.2 for A+B 18.8 for A-B 24.86 23.14 for C+D 15.42 16.58 for C-D

DUPLICATES:

Number of Data Pairs	C	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)

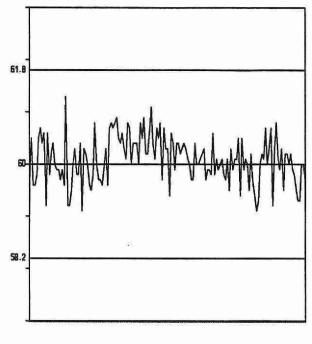
211	0.00	•	2.00	0.0421	10.3
51	2.00	•	5.00	0.0673	4.5
48	5.00	-	10.00	0.1100	14.7
57	10.00		20.00	0.2406	2.3
18	20.00	•	50.00	0.3625	1.2
385	(Overall		0.0842	

	Number of Data	Data Mean	Standard(1) Deviation

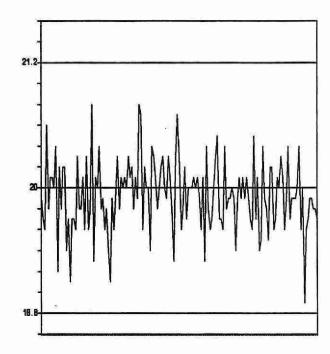
Long Term Blank	149	-0.003	0.044

NITROGEN, NITRATE PLUS NITRITE (mg/L as N)

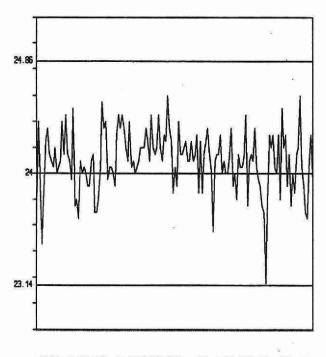
QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90



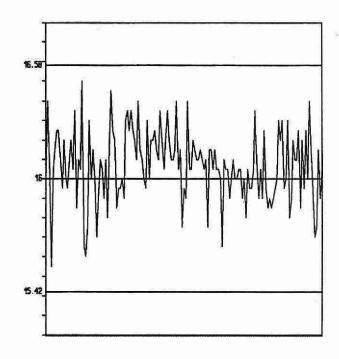
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** NITROGEN, NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory

: Colourimetry

Method Introduced

: 01/04/76

LIS Test Name Code Work Station Code

: NNOTUR : WFNO3 Units
Unit Code

: mg/L as N : 064807

Method Code

: 002CC2

Supervisor

: M. Rawlings

Sample Type/Matrix

: Ministry of Health Water Samples

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38°C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step. Approximate absorbance: 0.5 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37°C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.1

T value: 0.5

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration

: 2 standards, e.g. OCA

Drift

: BL every 10 samples; standard every 20 samples

Interference

: Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms

effective interference suppression.

Recovery

: Individual nitrate and nitrite standards of equal N concentration show effectiveness of

reduction step.

NITROGEN, NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 20.0 mg/L as N

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias .	Deviation
	***********	*******			
A :	109	16.0	16.094	0.094	0.105
B :	109	8.0	8.069	0.069	0.075
A+B:	109	24.0	24.163	0.163	0.144
A-B:	109	8.0	8.026	0.026	0.113
C :	109	8.0	8.069	0.069	0.075
D:	109	1.6	1.602	0.002	0.013
C+D:	109	9.6	9.671	0.071	0.078
C-D:	109	6.4	6.467	0.067	0.075

s.d.(AB) S(between runs): 0.09 Sw(within run): 0.08 S/Sw: 1.14

s.d.(CD) S(between runs): 0.054

Sw(within run): 0.052 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

25.0 23.0 for A+B 7.3 8.7 10.05 for 9.15 for C+D 5.95 6.85 for C-D

DUPLICATES:

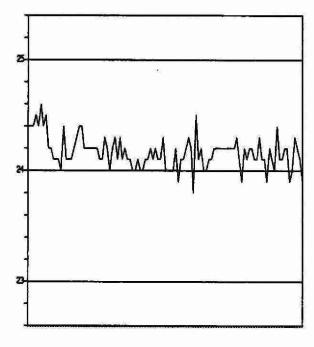
Number of	Sample			Mean(2)	Coefficient
Data Pairs	Concn Span		s.d.	of var.(%)	
	****		*****	*********	
182	0.00	•	4.00	0.052	4.8
35	4.00		10.00	0.106	1.7
15	10.00	•	20.00	0.233	2.3
232		Overal	1	0.101	

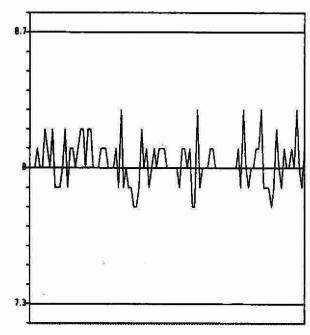
	Number	Data	Standard(1)
*	of Data	Mean	Deviation

Long Term Blank	109	0.0	0.0

NITROGEN, NITRATE PLUS NITRITE (mg/L as N)

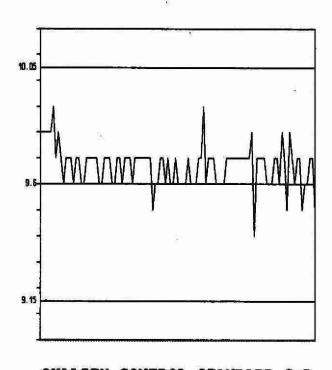
QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

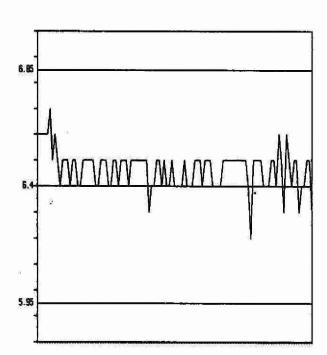




QUALITY CONTROL STANDARD A+B







QUALITY CONTROL STANDARD C-D

QUALITY CONTROL STANDARD C+D

*** NITROGEN, NITRITE ***

IDENTIFICATION:

Laboratory
LIS Test Name Code

: Colourimetry : NNO2FR Method Introduced Units : 01/04/78 : mg/L as N

Work Station Code Method Code : RNDNP : 102DC2

Unit Code Supervisor : 064807 : M. Rawlings

Sample Type/Matrix

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents

SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.6 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.001

T value: 0.005

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. OCA

Drift

: BL every 10 samples; standard every 20 samples

Interference

: Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms

effective interference suppression.

Recovery

: Individual nitrate and nitrite standards of equal N concentration show effectiveness of

reduction step.

NOTES:

1990 graph of quality control standard C-D showed the lower control limit was exceeded, and which fell below the QCC value. The data was reported and qualified with a warning remark.

NITROGEN, NITRITE

QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 0.200 mg/L as N

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	********			*******	
A :	119	0.16	0.1594	-0.0006	0.0013
B :	119	0.08	0.0796	-0.0004	0.0010
A+B:	119	0.24	0.2390	-0.0010	0.0019
A-B:	119	0.08	0.0798	-0.0002	0.0014
C :	119	0.08	0.0796	-0.0004	0.0010
D:	119	0.016	0.0159	-0.0001	0.0006
C+D:	119	0.096	0.0954	-0.0006	0.0013
C-D:	119	0.064	0.0637	-0.0003	0.0011

s.d.(AB) S(between runs): 0.0012

Sw(within run): 0.0010 S/Sw: 1.18

s.d.(CD) S(between runs): 0.0008

Sw(within run): 0.0007 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.231	18	0.249	for	A+B
0.074	•	0.086	for	A-B
0.092		0.100	for	C+D
0.061		0.067	for	C-D

DUPLICATES:

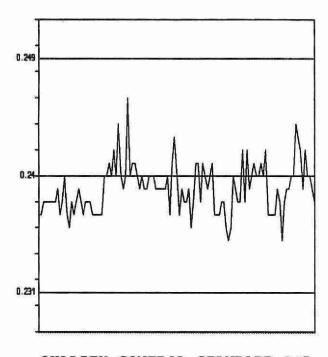
Number of Data Pairs	Sample Conon Span		Mean(2) s.d.	Coefficient of var.(%)	

183	0.000		0.005	0.0010	43.9
97	0.005		0.020	0.0017	24.2
33	0.020		0.200	0.0016	4.5
313	0	veral	1	0.0012	, and the second

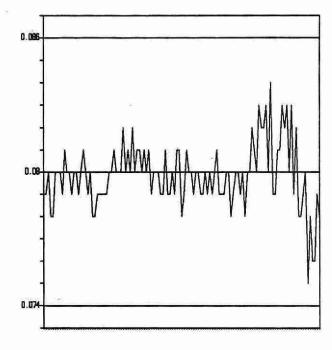
	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	119	-0.00000	0.00089

NITROGEN, NITRITE (mg/L as N)

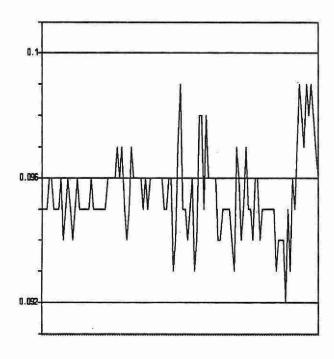
QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90



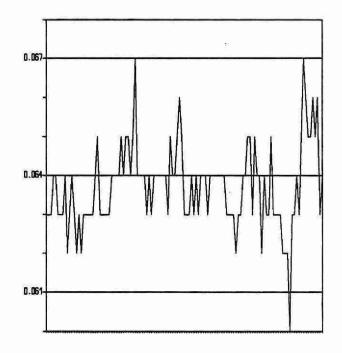
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** NITROGEN, NITRITE ***

IDENTIFICATION:

Laboratory

: Colourimetry LIS Test Name Code

Method Introduced : NNO2FR Units

: 01/04/78 : mg/L as N

Work Station Code

: SDNP : 102CC2 Unit Code Supervisor

: 064807 : M. Rawlings

Method Code Sample Type/Matrix

: Sewage, Industrial Waste, Leachate, Domestic Waters

SAMPLING:

Ouantity Required

: 10 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide, and N(1-napthyl) ethylenediamine dihydrochloride.

Approximate absorbance: 0.3 at the full scale level.

Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.005

T value: 0.025

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. OCA

Drift

: BL every 10 samples; standard every 20 samples

Interference

: Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms

effective interference suppression.

Recovery

: Individual nitrate and nitrite standards of equal N concentration show effectiveness of

reduction step.

NITROGEN, NITRITE

QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90

Lab: Colourimetry

Analytical Range: - to 2.0 mg/L as N

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
N/	of Data	Concn	Measured	Bias	Deviation
	**********	**********			
A :	153	1.6	1.5997	-0.0003	0.0104
B :	153	0.8	0.8021	0.0021	0.0061
A+B:	153	2.4	2.4018	0.0018	0.0120
A-B:	153	0.8	0.7975	-0.0025	0.0121
C :	153	0.8	0.8021	0.0021	0.0061
D:	153	0.16	0.1604	0.0004	0.0027
C+D:	153	0.96	0.9625	0.0025	0.0072
C-D:	153	0.64	0.6417	0.0017	0.0060

s.d.(AB) S(between runs): 0.0085

Sw(within run): 0.0086 S/Sw: 0.99

s.d.(CD) S(between runs): 0.0047

Sw(within run): 0.0042 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31	•	2.49	for	A+B
0.74	-	0.86	for	A-B
0.92	+	1.00	for	C+D
0.61	•	0.67	for	C-D

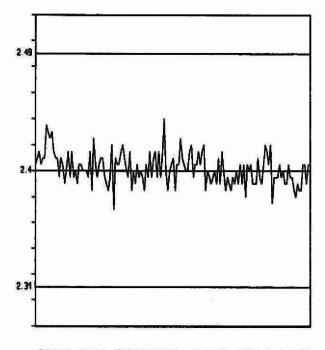
DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
274	0.00	-	0.20	0.004	33.9
50	0.20		1.00	0.021	7.3
11	1.00	-	2.00	0.085	3.3
445	(Overall		0.004	

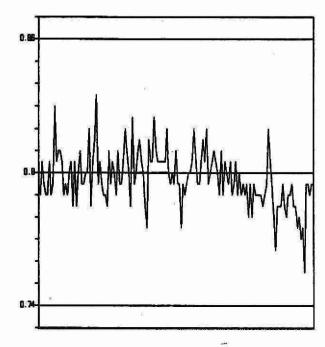
	Number of Data	Data Mean	Standard(1) Deviation
	****	*****	
Long Term Blank	153	-0.00003	0.0024

NITROGEN, NITRITE (mg/L as N

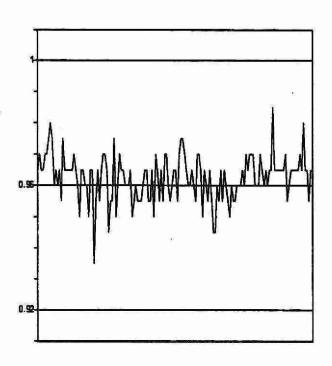
QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90



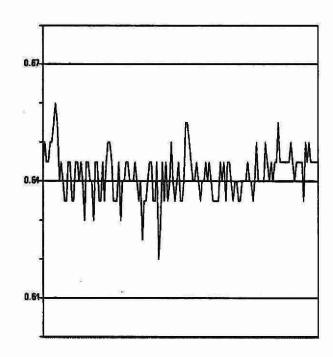
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** NITROGEN, TOTAL KJELDAHL ***

IDENTIFICATION:

Laboratory

: Colourimetry

Method Introduced

: 01/04/79

LIS Test Name Code

: NNTKUR : RTNP Units
Unit Code

: mg/L as N : 064807

Work Station Code Method Code

: 004AC2

Supervisor

: M. Rawlings

Sample Type/Matrix

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.3 at the full scale level. Total phosphorus is determined simultaneously.

INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Coulourimetric measurement is through a 5.0 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration

: LTBL plus 3 undigested standards, e.g. QCA

Recovery Drift : 3 digested BL plus 3 digested standards in duplicate, e.g. R1 : BL every 10 samples; undigested standard every 20 samples

NITROGEN, TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 2.0 mg/L as N

CALIBRATION CONTROL:

*	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			*********	*********	
A :	174	1.6	1.605	0.005	0.0187
B :	174	0.8	0.803	0.003	0.0098
A+B:	174	2.4	2.408	0.008	0.0227
A-B:	174	0.8	0.803	0.003	0.0196
C :	174	0.8	0.803	0.003	0.0098
D:	174	0.16	0.170	0.010	0.0078
C+D:	174	0.96	0.973	0.013	0.0149
C-D:	174	0.64	0.632	-0.008	0.0097

s.d.(AB) S(between runs): 0.015

Sw(within run): 0.014 S/Sw: 1.08

s.d.(CD) S(between runs): 0.009

Sw(within run): 0.007 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31	•	2.49	for	A+B
0.74	7 	0.86	for	A-B
0.92	1. E	1.00	for	C+D
0.616	•	0.664	for	C-D

RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard(1) Deviation

R1:	174	1.40	1.396	0.0546
R2:	174	0.84	0.829	0.0487
R3:	174	0.28	0.280	0.0240

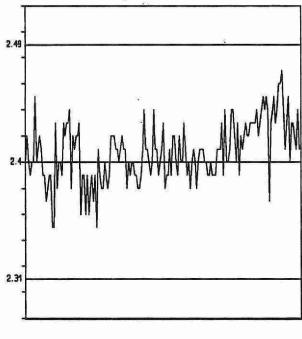
DUPLICATES:

Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
**********	*************		*********		
320	0.00	-	0.40	0.0222	14.7
148	0.40	***	1.00	0.0343	6.8
29	1.00		2.00	0.0403	3.5
497	Overall			0.0270	

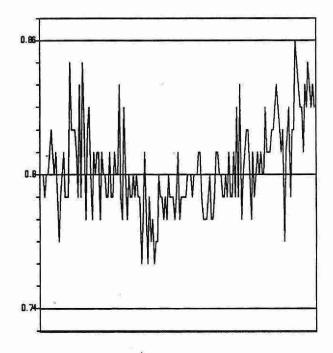
	Number of Data	Data	Standard(1)
	of Data	Меап	Deviation
	*****	*********	*********
Long Term Blank	174	-0.004	0.0082
Digested Blank	174	0.023	0.0182

NITROGEN, TOTAL KJELDAHL (mg/L as N)

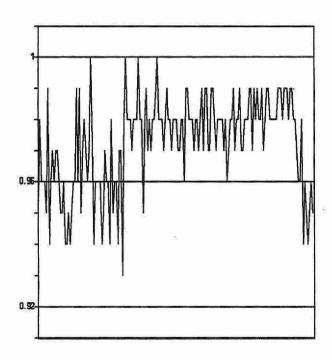
QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90



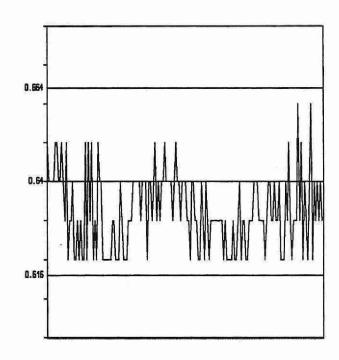
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

*** NITROGEN, TOTAL KJELDAHL ***

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code Method Code

: Colourimetry : NNTKUR

: 004BC2

Method Introduced Units : STKNP

Unit Code Supervisor

: 064807 : M. Rawlings

: 01/04/79

: mg/L as N

Sample Type/Matrix : Sewage, Industrial Waste, Domestic Waters, Effluents, Leachates

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digesters kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 1.1 at the full scale level. Total phosphorus is determined simultaneously.

INSTRUMENTATION:

Three block digesters

Basic automated modular continuous flow system plus 1 module: 38°C bath (7.7 mL delay). Coulourimetric measurement is through a 1.5 cm. light path at 630 nm. Data capture, reduction and processing via a multistage microcomputer system.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

CALIBRATION:

BL plus 7 undigested standards

CONTROLS:

Calibration

: LTBL plus 3 undigested standards, e.g. QCA

Recovery Drift

: 3 digested BL plus 3 digested standards in duplicate, e.g. R1 : BL every 10 samples; undigested standard every 20 samples

NOTES:

**System is calibrated with undigested standards.

NITROGEN, TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90

Lab: Colourimetry

Analytical Range: - to 50.0 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		********	**********	*********	***********
A :	200	40.0	40.066	0.066	0.190
B :	200	20.0	20.018	0.018	0.107
A+B:	200	60.0	60.084	0.084	0.249
A-B:	200	20.0	20.048	0.048	0.182
C :	200	20.0	20.018	0.018	0.107
D :	200	4.0	3.999	0.001	0.045
C+D:	200	24.0	24.017	0.017	0.125
C-D:	200	16.0	16.019	0.019	0.106

s.d.(AB) S(between runs): 0.15

Sw(within run): 0.13 S/Sw: 1.20

s.d.(CD) S(between runs): 0.08

Sw(within run): 0.75 S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

57.75	486	62.25	for	A+B
18.5	•	21.5	for	A-B
23.1	.44	24.9	for	C+D
15.4		16.6	for	C-D

RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard(1) Deviation

R1:	200	35.0	34.53	1.145
R2:	200	21.0	20.76	1.106
R3:	200	7.0	6.96	0.305

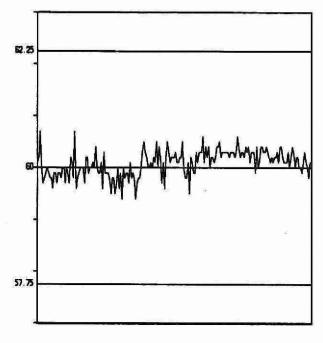
DUPLICATES:

Number of Data Pairs			Mean(2) s.d.	Coefficient of var.(%)	
*********					***************************************
173	0.00		0.50	0.0396	30.8
205	0.50	47	2.00	0.0855	14.4
100	2.00		10.00	0.1652	4.8
89	10.00	•	50.00	0.2997	2.6
567		Overall		0.1040	

	Number of Data	Data Mean	Standard(1) Deviation
×			
Long-Term Blank Digested Blank	200 200	0.0015 0.0320	0.026 0.060

NITROGEN, TOTAL KJELDAHL (mg/L as N)

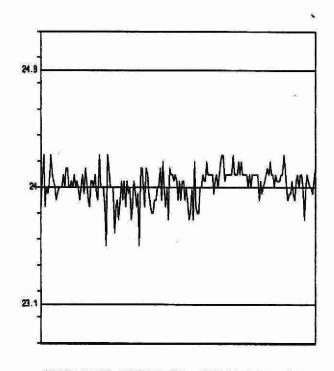
QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90

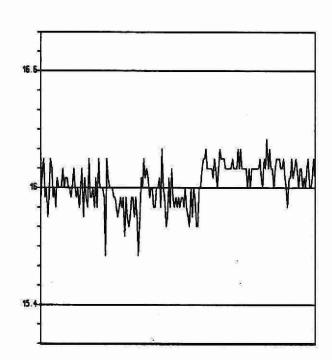


21.5 20.5 21.5 20.5 21.5 21.5 21.5

QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B





QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

_____CONTROL LIMIT

OXYGEN DEMAND, BIOCHEMICAL

IDENTIFICATION:

Laboratory

: Solids and BOD : BOD5

Method Introduced

: Before '61

LIS Test Name Code Work Station Code

: SBBOD5

Units Unit Code

: mg/L as O : 064808

Method Code Sample Type/Matrix : 001AI2

Supervisor

: P. Campbell

: Sewage, Industrial Waste, Effluents, Domestic Waters, Leachates

SAMPLING:

Ouantity Required

: 400 mL

Container

: Glass or plastic

SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

ANALYTICAL PROCEDURE:

Oxygen depletion is measured as the difference in dissolved oxygen (DO) concentration, DO readings are taken prior to sample storage, and also at the end of storage in the dark at 20°C for five days (BOD5). If necessary, dilutions are made with aerated, nutrient-enriched water to obtain a 25-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples, calculation of an appropriate seed correction is required.

INSTRUMENTATION:

-Weston and Stack Oxygen analyzer with DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen.

-Titration equipment for Winkler analysis of dissolved oxygen.

-Incubator (19-21°C); BOD bottles (300 mL)

REPORTING: .

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION (DO):

Blank is a sulphite solution (negligible DO) and the standard is air-saturated distilled, deionized water. The DO content of the latter is read from a table after measuring its temperature and the barometric pressure in the laboratory.

CONTROLS:

Calibration (DO)

: 2 OC solutions of distilled water which have been partially stripped of DO by flushing with nitrogen.

These "solutions", of different but unknown DO, are analyzed with the Oxygen Analyzer and by the Winkler titration procedure. The difference between the values for the two analytical methods is

utilized as a slope control for the DO Analyzer.

Recovery (BOD5)*

: 3 Recovery standards prepared from a combination of Glucose and Glutamic Acid e.g. R1; the

expected BOD5 is 67% of the oxygen requirement for complete oxidation.

Drift

: Air saturated distilled water after every 24 samples.

Blanks'

: Distilled deionized water and BOD dilution water

NOTES:

Currently tests on sewage are performed by a private laboratory, OC results reported here represent only tests performed at the Central Laboratory.

These solutions are incubated for five days alongside samples.

OXYGEN DEMAND, BIOCHEMICAL

QUALITY CONTROL DATA FROM 03/01/90 TO 28/12/90

Lab: Solids and BOD

Analytical Range: - to 400.0 mg/L as O

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	(man-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1	*********	* * * * * * * * * * * * * * * * * * * *		
A :	92	0.00	0.01	0.01	0.09
B:	92	0.00	0.00	0.00	0.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

-0.25

0.25

RECOVERIES:

	Number	Expected	Av. Concn	Standard(1)
	of Data	Concn	Measured	Deviation
R1:	153	2.20	2.16	0.10
R2:	152	4.34	4.27	0.19
R3:	151	6.52	6.34	0.32

DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
82	0	-	5	0.12	7.40
15	5	/ 10 00	20	0.44	4.38
5	20		100	2.37	7.59
8	100	-	400	7.70	3.29
110		Overal	1	0.21	UNION TO V

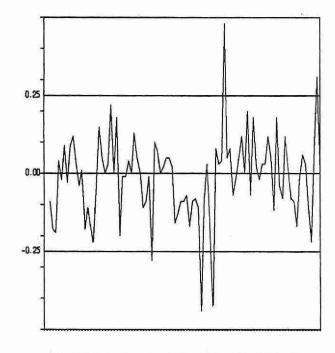
OTHER CHECKS:

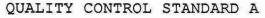
	Number	Data	Standard(1)
	of Data	Mean	Deviation

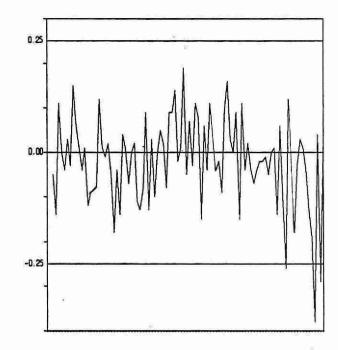
5 Day DDW Blank	155	0.14	0.10
5 Day BOD Blank	154	0.14	0.09

OXYGEN DEMAND, BIOCHEMICAL (mg/L as 0)

QUALITY CONTROL DATA FROM 03/01/90 TO 28/11/90







QUALITY CONTROL STANDARD B

_____CONTROL LIMIT

*** OXYGEN DEMAND, CHEMICAL ***

IDENTIFICATION:

Laboratory

...

: Colourimetry : COD Method Introduced

: 01/07/82

LIS Test Name Code Work Station Code

: RCOD

Unit Code

Units

: mg/L as O : 064808

Method Code

: 5251C2

Supervisor

: M. Rawlings

Sample Type/Matrix

: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required

: 25 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.05 at the full scale level.

INSTRUMENTATION:

-Culture tubes with Teflon closures; mechanical-convection oven

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3

Current W value: 1

T value: 5

CALIBRATION:

3 digested BL plus 3 digested standards

CONTROLS:

Calibration

: 2 digested standards, e.g. QCA

Recovery

: 2 digested standards, e.g. R1

Drift

: Undigested BL every 10 samples; standard plus BL at end of run

Interference

: Digested standard (40 mg/L as O) spiked with 50 mg/L Cl confirms suppression of

chloride interference.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week.

OXYGEN DEMAND, CHEMICAL

QUALITY CONTROL DATA FROM 04/01/90 TO 18/12/90

Lab: Colourimetry

Analytical Range: - to 40.0 mg/L as O

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	******	********	**********	***********	
A :	73	40.0	39.994	-0.006	0.658
B :	73	10.0	10.017	0.017	0.711
A+B:	73	50.0	50.011	0.011	1.061
A-B :	73	30.0	29.977	-0.023	0.865

s.d.(AB) S(between runs): 0.68 Sw(within run): 0.61 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 53.7 46.3 for A+B 27.2 32.8 for A-B

RECOVERIES:

Number of Data		Expected Concn	Av. Concn Measured	Standard(1) Deviation	
	400000000			*********	
R1:	73	39.0	35.13	2.77	
R2:	73	9.8	8.97	2.03	

DUPLICATES:

Number of Data Pairs	C	Sample Concn S		Mean(2) s.d.	Coefficient of var.(%)

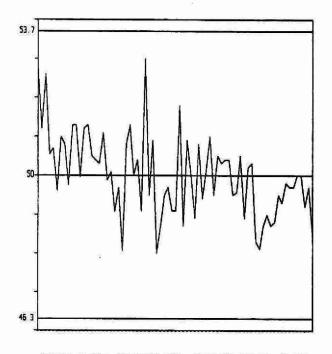
32	0.0	<u> </u>	10.0	1.12	16.7
63	10.0	-	25.0	1.52	10.1
33	25.0		50.0	1.72	5.1
128		Overall		1.46	

OTHER CHECKS:

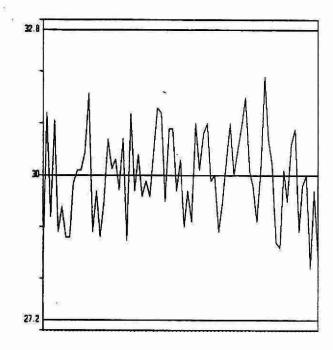
	Number	Data	Standard(1)
	of Data	Mean	Deviation
Chloride Check	73	39.58	3.79

OXYGEN DEMAND, CHEMICAL (mg/L as 0)

QUALITY CONTROL DATA FROM 04/01/90 TO 18/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** OXYGEN DEMAND, CHEMICAL ***

IDENTIFICATION:

Laboratory

: Colourimetry

Method Introduced

: 01/07/82 : mg/L as O

LIS Test Name Code Work Station Code

: COD : SBCOD Units Unit Code

: 064808

Method Code

: 002AC0

Supervisor

: M. Rawlings

Sample Type/Matrix

: Sewage, Industrial Waste, Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required

: 25 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150°C. Analysis is completed by automated colourimetric measurement of trivalent chromium. Approximate absorbance: 0.6 at the full scale level.

INSTRUMENTATION:

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3

Current W value: 2

T value: 10

CALIBRATION:

2 digested BL plus 4 digested standards

CONTROLS:

Calibration

: 2 digested standards, e.g. QCA

Recovery Drift

: 2 digested standards, e.g. R1 : Undigested BL every 10 samples; standard plus BL at end of run

Interference

: Digested standard (50 mg/L as O) spiked to 900 mg/L Cl confirms suppression of

chloride interference.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week. The recovery standard is a material known to be very difficult to digest. The expected recovery is approximately 85%, based on long term experience. We continue to use this material in spite of the poor recovery, because if the slightest problem exists with the digestion step, the recovery falls off sharply to approximately 10%.

OXYGEN DEMAND, CHEMICAL

QUALITY CONTROL DATA FROM 10/01/90 TO 28/12/90

Lab: Colourimetry

Analytical Range: - to 500.0 mg/L as O

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			********	*****	
A :	69	400	399.0	-1.0	4.89
B :	69	100	102.9	2.9	4.06
A+B:	69	500	501.9	1.9	7.59
A-B:	69	300	296.1	-3.9	4.92

s.d.(AB) S(between runs): 4.5 Sw(within run): 3.5 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5

522.5 315.0

A+B for

285.0

for A-B

RECOVERIES:

18	Number of Data	Expected Concn	Av. Concn Measured	Standard(1) Deviation
		**********		*********
R1:	69	390	383.3	58.2
R2:	69	98	96.1	6.3

DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*****				***************************************	
79	0		100	3.06	9.9
25	100	*	250	4.83	2.9
29	250	=	500	7.25	2.2
133	(Overa	1	4.30	

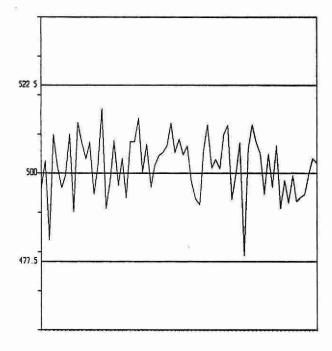
OTHER CHECKS:

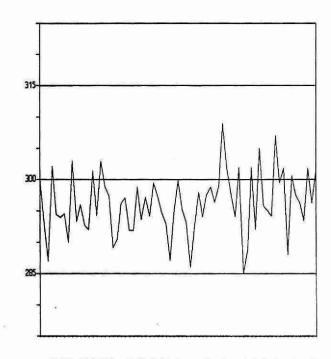
	Number	Data	Standard(1)
	of Data	Mean ⁻	Deviation

Chloride Check	66	58.7	6.82

OXYGEN DEMAND, CHEMICAL (mg/L as 0)

QUALITY CONTROL DATA FROM 10/01/90 TO 28/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** pH ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 01/01/76

LIS Test Name Code Work Station Code

: pH : DOCOP Units

: dimensionless

Method Code

: 0903PH

Unit Code

: nil

Sample Type/Matrix

: Lakes

Supervisor

: A. Neary

SAMPLING:

Quantity Required

: 100 mL

Container

: BOD bottle filled to the brim; screw caps with cone-shaped liners.

ANALYTICAL PROCEDURE:

pH is measured directly on a stirred sample (50 mL) at room temperature by a pH meter. Stirring rate, beaker size, degree of electrode immersion and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

Digital pH meter, stirrer, combined glass electrode.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration

: BL plus 2 standards, e.g. QCA, QCB

Drift

: 2 standard buffers - 2 times daily

pH

QUALITY CONTROL DATA FROM 09/01/90 TO 04/12/90

Lab: Dorset

Analytical Range: - to 14.00 Dimensionless

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	53	6.86	6.859	-0.001	0.136
B :	53	4.00	4.043	0.043	0.046
A+B:	53	10.86	10.902	0.042	0.138
A-B:	53	2.86	2.816	-0.044	0.150

s.d.(AB) S(between runs): 0.102

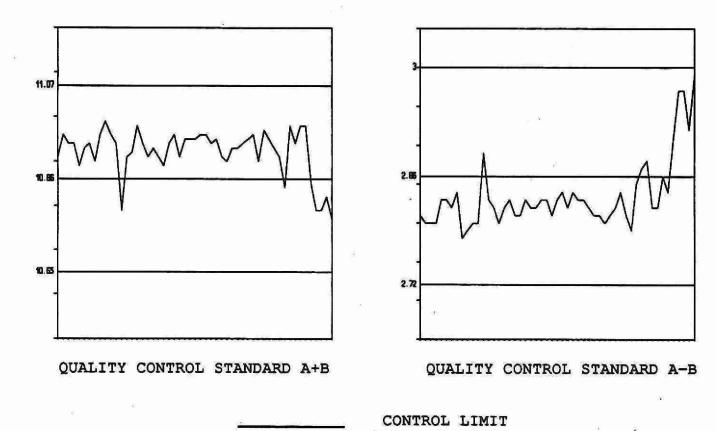
Sw(within run): 0.106 S/Sw: 0.96

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 - 11.07 for A+B 2.72 - 3.00 for A-B

Number of Data Pairs		Samponen	ole Span	Mean(2) s.d.	Coefficient of var.(%)
26	3.50	-	5.50	0.021	0.4
95	5.50	-	6.50	0.041	1.1
23	6.50		7.20	0.034	0.5
0	7.2	-	14.00	N.A.	N.A.
144	C)vera	11	0.035	

PH
QUALITY CONTROL DATA FROM 09/01/90 TO 04/12/90



- 252 -

pH ***

IDENTIFICATION:

Laboratory

: Dorset

Method Introduced

: 01/01/76

LIS Test Name Code

: pH : DOT Units

: dimensionless

Work Station Code

Unit Code

: nil

Method Code Sample Type/Matrix : 0902PH

Supervisor : Streams, Lakes, Precipitation, and Groundwater : A. Neary

Quantity Required

:150 mL

Container

SAMPLING:

:250 mL Amber polyethylene or BOD bottle filled to the brim; screw caps with

cone-shaped liners are preferred.

ANALYTICAL PROCEDURE:

pH is measured directly on a stirred sample (100 mL) at room temperature. Stirring rate, beaker size, degree of electrode immersion and room temperature range are uniform for all samples and standards. Alkalinity (Gran) is performed simultaneously.

INSTRUMENTATION:

Digital pH meter, stirrer, combined glass electrode.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration

: BL plus 2 standards, e.g. QCA, QCB

Drift

: 2 standard buffers - 2 times daily

QUALITY CONTROL DATA FROM 04/01/90 TO 17/12/90

Lab: Dorset

Analytical Range: - to 14.00 Dimensionless

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			**********		*********
A :	215	6.86	6.875	0.015	0.017
B :	215	4.00	4.038	0.038	0.032
A+B:	215	10.86	10.913	0.053	0.030
A-B :	215	2.86	2.837	-0.023	0.042

s.d.(AB) S(between runs): 0.026

Sw(within run): 0.029 S/Sw: 0.87

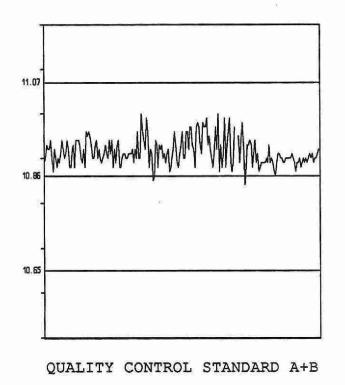
On any given day the calibration is accepted if the values obtained lie within the ranges:

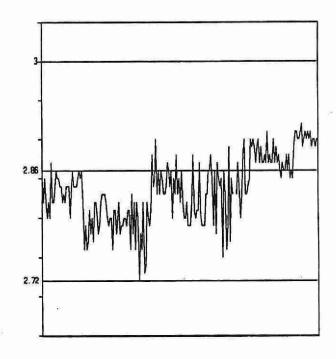
10.65 - 11.07 for A+B 2.72 - 3.00 for A-B

Number of Data Pairs		ample non Sp		Mean(2) s.d.	Coefficient of var.(%)	

172	3.5	*	5.0	0.019	0.5	
226	5.0	-	6.0	0.045	1.3	
155	6.0	=	7.0	0.047	0.7	
9	7.0		9.5	0.073	0.9	
562	()veral	1	0.039		

pH
QUALITY CONTROL DATA FROM 04/01/90 TO 17/12/90





QUALITY CONTROL STANDARD A-B

_____ CONTROL LIMIT

*** pH ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: 01/05/79

LIS Test Name Code Work Station Code : PH : PHACD Units Unit Code

: Dimensionless

Method Code

: 002AII

Supervisor

; nil : F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint acidity and Gran acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

pН

QUALITY CONTROL DATA FROM 02/01/90 TO 12/12/90

Lab: Titration

Analytical Range: - to 14.00 Dimensionless

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	104	4.45	4,479	0.029	0.0359
B :	104	3.75	3.765	0.015	0.0298
A+B : A-B :	104 104	8.20 0.70	8.244 0.714	0.044 0.014	0.0554 0.0357

s.d.(AB) S(between runs): 0.033

Sw(within run): 0.025 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

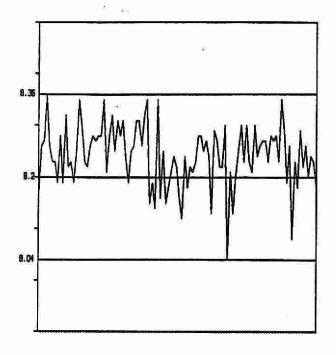
8.04 - 8.36 for A+B 0.59 - 0.81 for A-B

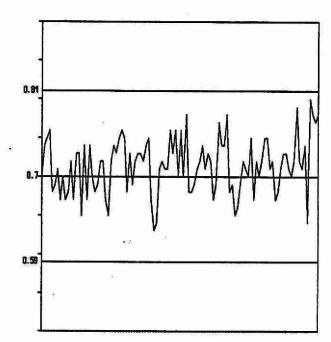
Number of	Sample			Mean(2)	Coefficient
Data Pairs	Concn Span		s.d.	of var.(%)	

26	3.5		4.0	0.0182	0.5
11	4.0	•	5.0	0.0294	0.8
34	5.0	ça.	8.5	0.0906	2.6
171	(Overal	1	0.0337	

PH

QUALITY CONTROL DATA FROM 02/01/90 TO 12/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

_____CONTROL LIMIT

pH

IDENTIFICATION:

Laboratory

: Titration

: PH

Method Introduced Units

: 09/07/80

LIS Test Name Code Work Station Code

: RATS

Unit Code

: Dimensionless : nil

Method Code Sample Type/Matrix : 003AI2 : Rivers, Lakes Supervisor

: F. Lo

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Gran Alkalinity, total fixed endpoint alkalinity, and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range 4 to 9

CONTROLS:

Calibration

: 2 QC standards at ph 7.41 and pH 4.45 e.g. QCA

Drift

: In run standards throughout the run (diluted tap water 20% V/V)

pH

QUALITY CONTROL DATA FROM 03/01/90 TO 27/12/90

Lab: Titration

Analytical Range: - to 14.00 Dimensionless

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
e e	*********		***********		************
A :	79	7.41	7.436	0.026	0.0331
B :	79	4.45	4.500	0.050	0.0480
A+B:	79	11.86	11.937	0.077	0.0641
A-B :	79	2.96	2.936	-0.024	0.0520

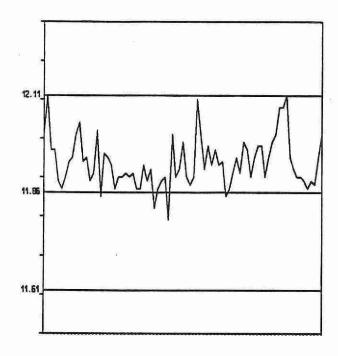
s.d.(AB) S(between runs): 0.041 Sw(within run): 0.037 S/Sw: 1.12

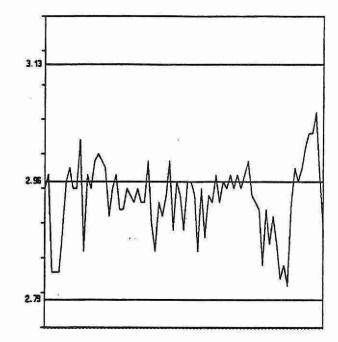
On any given day the calibration is accepted if the values obtained lie within the ranges:

11.61 2.79 12.11 for A+B 3.13 for A-B

Number of Data Pairs		Samp oncn S		Mean(2) Coes on s.d. of v	
********	***	*****			
27	3.50	-	7.00	0.1676	5.4
81	7.00		8.00	0.0896	1.2
114	8.00	-	9.50	0.0827	1.4
222	C)veral	Ĺ	0.0949	75.

pH
QUALITY CONTROL DATA FROM 03/01/90 TO 27/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** pH ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: 09/07/80

LIS Test Name Code Work Station Code : PH : WATS Units Unit Code : Dimensionless

Method Code

: 003A12

Supervisor

: nil : F. Lo

Sample Type/Matrix

: Domestic Waters, Sewage, Effluents

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Total fixed endpoint alkalinity and conductivity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range 4 to 9

CONTROLS:

Calibration

: 2 standards e.g. QCA

Drift

: In run standards throughout the run (tap water diluted to 50% V/V)v

pН

QUALITY CONTROL DATA FROM 02/01/90 TO 31/12/90

Lab: Titration

Analytical Range: - to 14.00 Dimensionless

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	105	7.41	7.44	0.03	0.0282
B :	105	4.45	4.49	0.04	0.0426
A+B:	105	11.86	11.94	0.08	0.0563
A-B:	105	2.96	2.95	-0.01	0.0453

s.d.(AB) S(between runs): 0.036

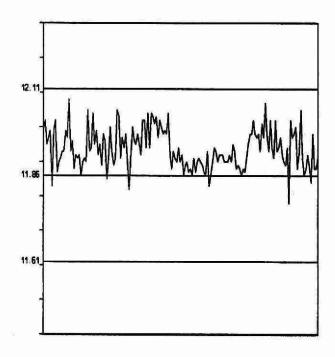
Sw(within run): 0.032 S/Sw: 1.13

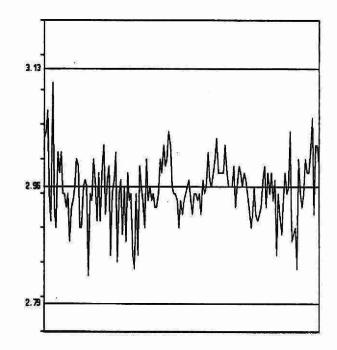
On any given day the calibration is accepted if the values obtained lie within the ranges:

11.61 - 12.11 for A+E 2.79 - 3.13 for A-B

Number of Data Pairs	C	Samponen :		Mean(2) s.d.	Coefficient of var.(%)
	*****				***********
27	2.00		7.00	0.2041	3.3
230	7.00		8.00	0.1466	1.9
151	8.00	-	9.00	0.0935	1.2
14	9.00	-	12.50	0.1253	1.4
0	12.50	e.	14.00	N.A	N.A
422)vera	1	0.1281	

PH
QUALITY CONTROL DATA FROM 02/01/90 TO 31/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

_____CONTROL LIMIT

*** Hq ***

IDENTIFICATION:

Laboratory

: Titration

Method Introduced

: Before '70

LIS Test Name Code

: PH : WQSDIRT Units

: Dimensionless

Work Station Code Method Code

: 004AI4

Unit Code Supervisor : Nil : F. Lo

Sample Type/Matrix

: Landfill leachates

SAMPLING:

Quantity Required

: 15 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (15 mL) at room temperature. Stirring rate and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

pH meter, stirrer, Radiometer combination electrode

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9

CONTROLS:

Calibration

: 2 standards e.g. QCA

QUALITY CONTROL DATA FROM 08/01/90 TO 17/12/90

Lab: Titration

Analytical Range: - to 14.00 Dimensionless

CALIBRATION CONTROL:

×	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	39	7.41	7.437	0.027	0.0278
B :	39	4.45	4.507	0.057	0.0455
A+B:	39	11.86	11.944	0.084	0.0494
A-B:	39	2.96	2.929	-0.031	0.0570

s.d.(AB) S(between runs): 0.038

Sw(within run): 0.040 S/Sw: 0.93

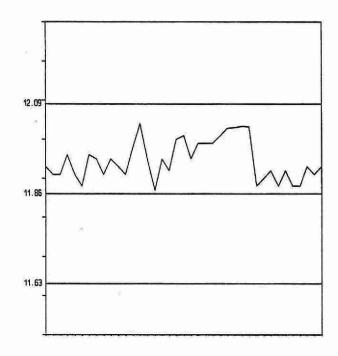
On any given day the calibration is accepted if the values obtained lie within the ranges:

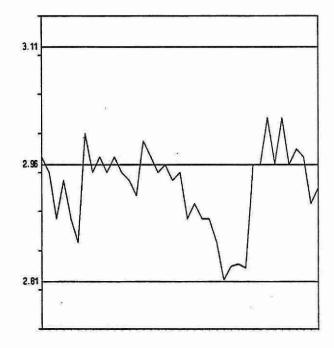
11.63 - 12.09 for A+B 2.81 - 3.11 for A-B

Number of Data Pairs		Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)

15	4.50	-	7.00	0.0291	0.5
22	7.00	•	8.00	0.0705	0.9
24	8.00	•	9.00	0.0570	0.7
61	C	veral	1	0.0546	

PH
QUALITY CONTROL DATA FROM 08/01/90 TO 17/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

_____ CONTROL LIMIT

*** pH ***

IDENTIFICATION:

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80 : dimensionless

LIS Test Name Code

: PHECA : DOSOILPH Units : din
Unit Code : nil

Work Station Code Method Code

: 324AB1

Supervisor

: A. Neary

Sample Type/Matrix

: Soil

SAMPLING:

Quantity Required

: 20 g dry

Container

: Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

Ten grams of sample (<2 mm) plus 20 mL 0.01 M calcium chloride are agitated in a tube for 20 minutes. The mixture is removed and allowed to equilibrate for 30 minutes. pH is measured on the supernatant.

INSTRUMENTATION:

-Corning pH/ion meter 150

-Corning Combination X-EL electrode balance accurate to 0.001 g.

REPORTING:

Maximum Significant Figures: 2

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration

: 3 buffers

Recovery

: 3 long term soil samples plus a round robin ECSS sample (latter run occasionally).

pH

QUALITY CONTROL DATA FROM 07/02/90 TO 13/11/90

Lab: Dorset Soils

Analytical Range: - to 10 Dimensionless

CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	*********			********	
. A :	25	7.0	6.998	-0.002	0.004
B:	25	4.0	4.004	0.004	0.004
A+B:	25	11.0	11.002	0.002	0.005
A-B:	25	3.0	2.994	-0.006	0.007
C :	25	6.8	6.776	-0.004	0.024
D:	25	4.0	4.004	0.004	0.004
C+D:	25	10.8	10.780	-0.020	0.026
C-D:	25	2.8	2.771	-0.009	0.024

s.d.(AB) S(between runs): 0.004

Sw(within run): 0.005 S/Sw: 0.86

s.d.(CD) S(between runs): 0.0175

Sw(within run): 0.0173 · S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.90		11.10	for	A+B
2.90	•	3.10	for	A-B
10.70		10.90	for	C+D
2.70	-	2.90	for	C-D

RECOVERIES:

	Number of Data	Av. Concn • Measured	Standard(1) Deviation

R1:	27	8.51	0.080
R2:	27	5.78	0.153
R3:	27	5.42	0.069

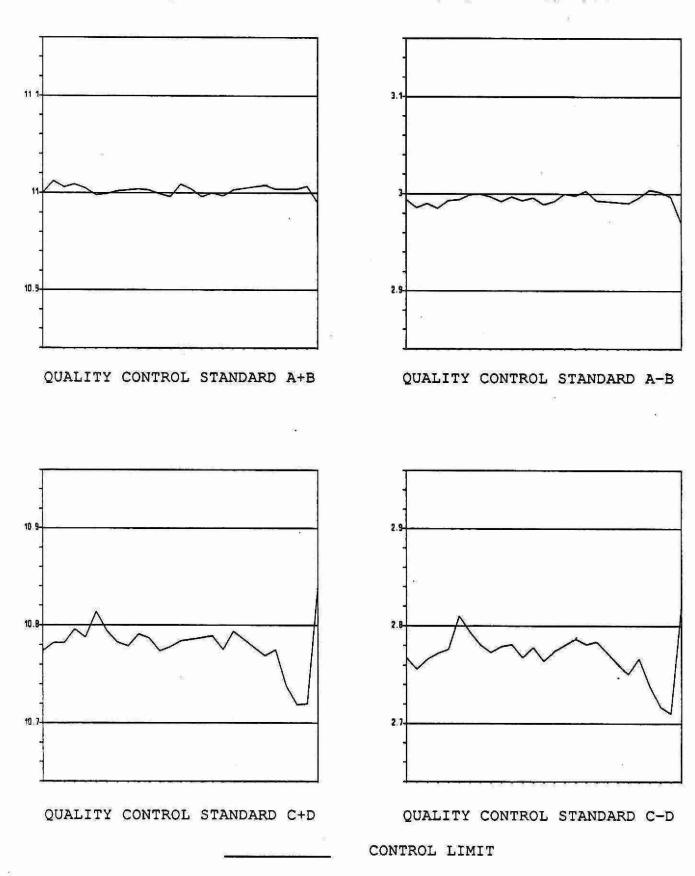
DUPLICATES:

Number of Data Pairs		Sample Concn Sp		Mean(2) s.d.	Coefficient of var.(%)
*********					**********
13	3.0	.	5.0	0.031	0.9
28	5.0		7.0	0.030	0.6
10	7.0	-	8.0	0.057	1.0
51		Overall		0.035	

OTHER CHECKS:

	Number of Data	Data Mean	Standard(1) Deviation
	*********	***********	***************************************
Slope	25	58.37	0.340

PH
QUALITY CONTROL DATA FROM 07/02/90 TO 13/11/90



*** pH ***

IDENTIFICATION:

Laboratory : Dorset Soils

Method Introduced : 01/06/80 Units : dimensionless

: A. Neary

LIS Test Name Code : PHEW
Work Station Code : DOSOILPH

Unit Code : nil

Method Code : 304AB1 Supervisor

Sample Type/Matrix : Soil

SAMPLING:

Quantity Required

: 20 g dry

Container

: Glass or plastic

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

Ten grams of sample (<2 mm) plus 20 mL of deionized water are agitated in a tube for 20 minutes. The mixture is removed and allowed to equilibrate for 30 minutes. pH is measured on the supernatant.

INSTRUMENTATION:

-Corning pH/ion meter 150

-Corning Combination X-EL electrode balance accurate to 0.001 g.

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

CONTROLS:

Calibration

:3 buffers

Recovery

:3 long term soil samples plus a round robin ECSS sample (run occasionally).

QUALITY CONTROL DATA FROM 07/02/90 TO 13/11/90

Lab: Dorset Soil	ills	S
------------------	------	---

Analytical Range: - to 10 Dimensionless

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	25	7.0	6.998	-0.002	0.004
B :	25	4.0	4.004	0.004	0.004
A+B:	25	11.0	11.002	0.002	0.005
A-B:	25	3.0	2.994	-0.006	0.007
C :	25	6.8	6.776	-0.004	0.024
D:	25	4.0	4.004	0.004	0.004
C+D:	25	10.8	10.780	-0.020	0.026
C-D:	25	2.8	2.771	-0.009	0.024

s.d.(AB) S(between runs): 0.004

Sw(within run): 0.005 S/Sw: 0.86

s.d.(CD) S(between runs): 0.0175

Sw(within run): 0.0173 S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.90	•	11.10	for	A+B
2.90	=	3.10	for	A-B
10.70		10.90	for	C+D
2.70	des.	2.90	for	C-D

RECOVERIES:

Number of Data		Av. Concn Measured	Standard(1) Deviation	
	*******		**********	********
R1:	25		7.59	0.203
R2:	25	*	4.93	0.105
R3:	25		4.82	0.045

DUPLICATES:

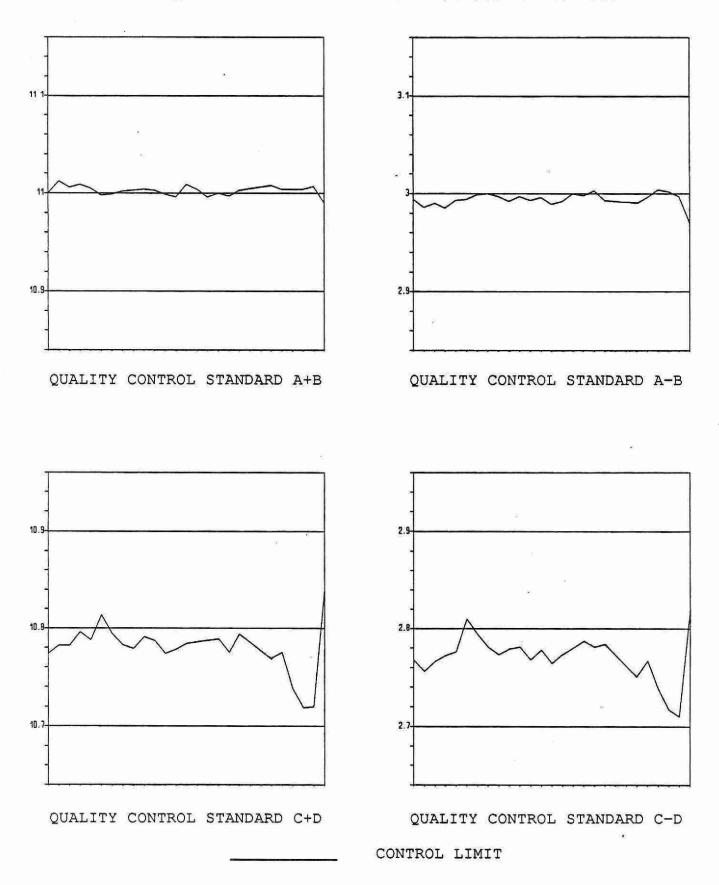
Number of Data Pairs		Sample Concn Sp	en	Mean(2) s.d.	Coefficient of var.(%)
				****	***********
8	3.0	*	4.0	0.020	0.5
30	4.0	8	5.0	0.016	0.4
26	5.0		8.0	0.038	0.6
64		Overall		0.025	17,245

OTHER CHECKS:

	of Data	Data Mean	Standard(1) Deviation

Slope	25	58.37	0.340

QUALITY CONTROL DATA FROM 07/02/90 TO 13/11/90



PHENOLICS, REACTIVE ***

IDENTIFICATION:

Laboratory

: MISA

Method Introduced

: 01/03/89

LIS Test Name Code

: PHNOL : MPHEN

Units Unit Code : ug/L as Phenol

Work Station Code Method Code

: 002BC3

Supervisor

: 063704 : P. Campbell

Sample Type/Matrix

: Sewage, Industrial Wastes, Leachates

SAMPLING:

Quantity Required

: 250 mL

Container

: Glass

ANALYTICAL PROCEDURE:

Samples are screened for oxidizing and reducing potential and neutralized, if required, and pH adjusted to 4 +/- 0.3 before manual distillation. Reactive phenolics are determined on the distillate colourimetrically, by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide. Approximate absorbance: 0.022 at full scale

INSTRUMENTATION:

Basic automated modular continuous flow system. Colourimetric measurement is through a 5.0 cm light path at 505 nm.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

Blank plus 4 standards

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: Bl, standard, Bl every 10 samples

Recovery

: Spiked blank and spiked sample every 10 samples

Blank

: Manual distillation blank checked before using every apparatus

Matrix

: Distillate from every sample is analyzed at 50% further dilution

NOTES:

The MPHEN work station was set up specifically for the difficult and variable matrices in MISA effluents.

MODIFICATIONS:

Minor modifications have been made to the procedure since starting in 1989: e.g. glassware washing and proofing improved, rinse waterline changed from plastic to glass where possible, concentration of calibration solutions was modified to cover analytical range better, and the in-line heater was removed.

PHENOLICS, REACTIVE

QUALITY CONTROL DATA FROM 12/01/90 TO 14/12/90

Lab: Colourimetry

Analytical Range: - to 40.0 ug/L as Phenol

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	46	40	40.27	0.27	0.9459
B :	46	10	9.89	-0.11	0.4036
A+B:	46	50	50.15	0.15	1.1583
A-B:	46	30	30.38	0.38	0.8795

s.d.(AB) S(between runs): 0.73 Sw(within run): 0.62 S/Sw: 1.2

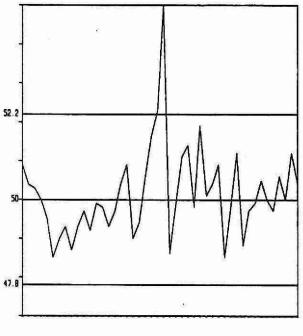
On any given day the calibration is accepted if the values obtained lie within the ranges:

52.2 47.8 for A+B 28.5 31.5 for A-B

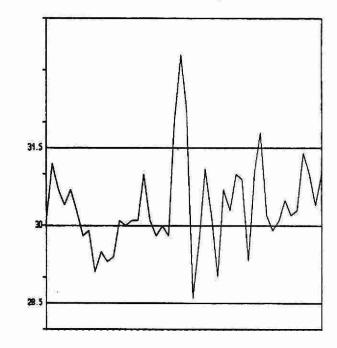
Number of	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
Data Pairs					
28	0.0	*	1.0	0.1153	16.5
29	1.0		5.0	0.2595	11.6
5	5.0	-	40.0	0.4591	11.9
62	Overall			0.1830	

PHENOLICS, REACTIVE (ug/L as Phenol)

QUALITY CONTROL DATA FROM 12/01/90 TO 14/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

*** PHENOLICS, REACTIVE ***

IDENTIFICATION:

Laboratory

: Colourimetry

Method Introduced

: 01/04/74

LIS Test Name Code

: PHNOL : ROPHEN

Units : u Unit Code : 0

: ug/L as Phenol : 063704

Work Station Code Method Code

: 002BC2

Supervisor

: M. Rawlings

Sample Type/Matrix

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies,

Leachates, Sewage, Industrial Wastes

SAMPLING:

Quantity Required

: 250 mL : Glass

Container Preservative

: Sulfuric acid to pH 1.5 - 2

Other

: Special bottle (with white cap) containing preservative is available

ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide.

Approximate absorbance: 0.03 at the full scale level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL, standard, BL every 10 samples

NOTES:

A report identifying reactive phenolics is available on request.

PHENOLICS, REACTIVE

QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 50.0 ug/L as Phenol

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	~~~~~~~	*********			
<b>A</b> :	115	40	40.10	0.10	0.340
B :	115	10	9.80	-0.20	1.547
A+B:	115	50	49.90	-0.10	0.794
<b>A-B</b> :	115	30	30.30	0.30	0.375

s.d.(AB) S(between runs): 0.39

Sw(within run): 0.34 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 - 52.2 for A+B 28.5 - 31.5 for A-B

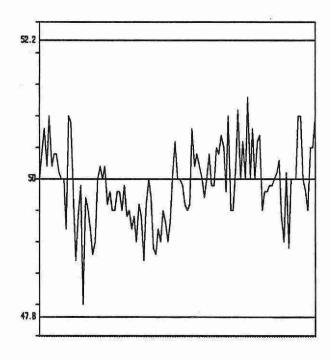
# **DUPLICATES:**

Number of Data Pairs				Mean(2) s.d.	Coefficient of var.(%)
*********				*******	
162	0.0	•	10.0	0.340	22.1
6	10.0	-	25.0	1.547	11.4
11	25.0		50.0	0.794	2.5
179		Overal		0.375	

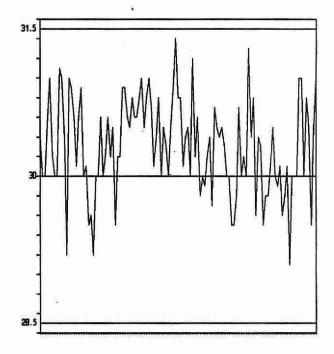
		Number	Data	Standard(1)
	H	of Data	Mean	Deviation
si .				***********
Long Term Blank	Ms	35	0.0001	0.0006

# PHENOLICS, REACTIVE (ug/L as Phenol)

QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

# *** PHOSPHORUS, BRAY II EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code Method Code

Sample Type/Matrix

: Dorset Soils

: PPO4BE : DOBEP

: 5926C3 : Soil

Method Introduced

Units Unit Code

: ug/g as P : 073815 Supervisor : A. Neary

: 1988

# SAMPLING:

Quantity Required

: 10 g air dried and sieved to < 2mm.

Container

: Glass or Plastic

# ANALYTICAL PROCEDURE:

A soil sample is weighed into centrifuge tubes. 25 ml of NH4F-HCl extractant is added and the tubes are capped and shaken for 1 hour. Samples are centrifuged and filtered through 0.45 um filters. The filtrate is analyzed by colourimetry.

#### INSTRUMENTATION:

Technicon colourimeter, peristaltic pump, sampler, and chart recorder.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

#### CALIBRATION:

6 standards covering the range 0 - 100 ug/g P

#### CONTROLS:

3 long term soil samples and 2 method blanks

QCA, QCB prepared by judiciously mixing previously analyzed samples; enough is prepared for 1 yr (1 litre)

# PHOSPHORUS, BRAY II EXTRACTABLE

# QUALITY CONTROL DATA FROM 08/01/90 TO 28/08/90

Lab: Dorset Soils

Analytical Range: - to 100.0 ug/g as P

# CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		**********		********	
A :	14	82.50	80.81	-1.69	2.023
B :	14	31.00	31.02	0.02	0.526
A+B:	14	113.50	111.83	-1.67	2.194
A-B:	14	51.50	49.79	-1.71	1.982

s.d.(AB) Sw(within run): 1.40

S(between runs): 1.48 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

106 - 121 for A+B 46.5 - 56.5 for A-B

#### RECOVERIES:

	Number	Av. Concn	Standard(1)
	of Data	Measured	Deviation
	****	~~~~~~~	
R1:	14	166.1	7.729
R2:	14	16.7	1.884
R3:	14	11.2	1.525

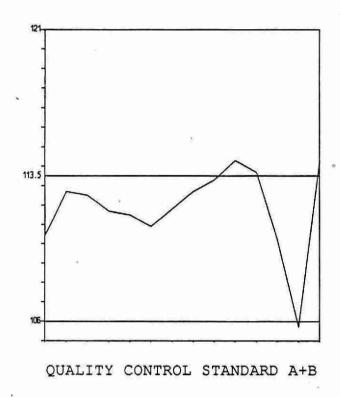
#### **DUPLICATES:**

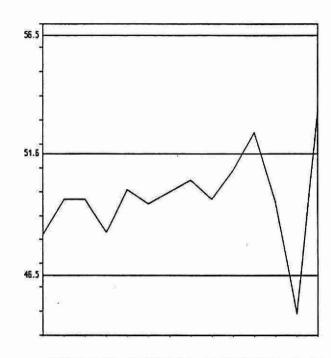
Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
					************
21	0.0		20.0	0.514	5.7
8	20.0	-	50.0	1.210	4.7
14	50.0	-	100.0	3.210	3.8
43	(	Overa	11	1.214	

	Number of Data	Data Mean	Standard(1) Deviation
		***********	
Method Blank	14	0.000	0.000

# PHOSPHORUS, BRAY II EXTRACTABLE (ug/g as P)

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90





QUALITY CONTROL STANDARD A-B

# *** PHOSPHORUS, REACTIVE ortho-PHOSPHATE ***

#### **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: 01/04/79

LIS Test Name Code Work Station Code

: PPO4FR : RNDNP

Units Unit Code

: mg/L as P : 064815

Method Code Sample Type/Matrix : 103DC2

Supervisor

: M. Rawlings

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents

#### SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.2 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.0005

T value: 0.0025

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

#### NOTES:

1990 incidents of control limits that were exceeded for the differences in the standards for A-B and C-D were shown to be due to contamination in the mid-range standard from an unidentified source. This unidentified contamination will force the W and T values to be doubled to 0.001 and 0.005 respectively for 1992 data, or until the problem is identified and controlled.

# PHOSPHOROUS, REACTIVE ortho-PHOSPHATE

# QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 0.10 mg/L as P

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	123	0.08	0.0798	-0.0002	0.0011
B:	123	0.04	0.0401	0.0001	0.0008
A+B:	123	0.12	0.1199	-0.0001	0.0016
A-B:	123	0.04	0.0397	-0.0003	0.0012
<b>C</b> :	123	0.04	0.0401	0.0001	0.0008
D:	123	0.008	0.0081	0.0001	0.0007
C+D:	123	0.048	0.0482	0.0002	0.0013
C-D:	123	0.032	0.0319	-0.0003	0.0008

s.d.(AB) S(between runs): 0.001

Sw(within run): 0.0008 S/Sw: 1.2

s.d.(CD) S(between runs): 0.0008

Sw(within run): 0.0006 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.115 - 0.125 for A+B 0.037 - 0.043 for A-B 0.045 - 0.051 for C+D 0.030 - 0.034 for C-D

#### **DUPLICATES:**

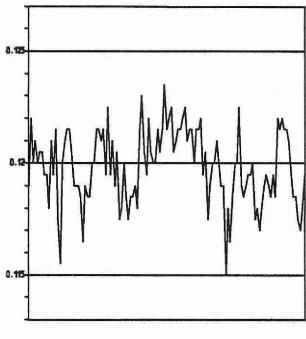
Number of Data Pairs				Mean(2) s.d.	Coefficient of var.(%)
*******					***************************************
197	0.00	-	0.02	0.0013	40.9
15	0.02		0.05	0.0035	12.0
7	0.05	-	0.10	0.0039	15.9
219	(	Overali	l.	0.0024	

	Number	Data	Standard(1)
	of Data	Mean	Deviation
	* *	***********	
Long Term Blank	123	0.0008	0.0086

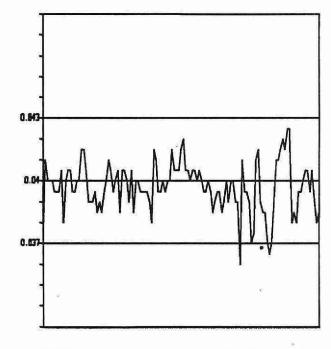
# PHOSPHORUS, REACTIVE ortho-PHOSPHATE

(mg/L as P)

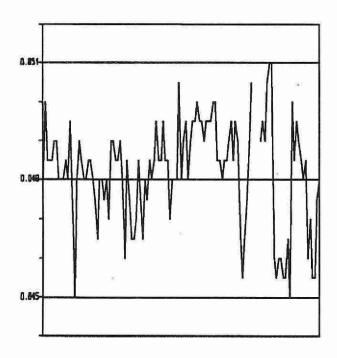
QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90



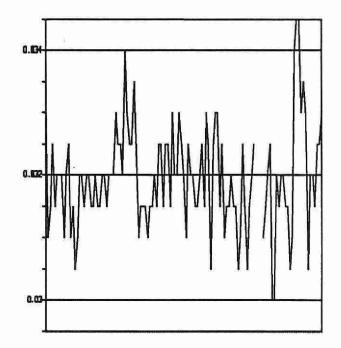
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B'



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D
CONTROL LIMIT

# *** PHOSPHORUS, REACTIVE ortho-PHOSPHATE ***

#### **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: 01/04/79

LIS Test Name Code

: PPO4FR : SDNP Units

: mg/L as P : 064815

Work Station Code Method Code

: 103BC2

Unit Code Supervisor

: M. Rawlings

Sample Type/Matrix

: Sewage, Industrial Waste, Leachate, Domestic Waters, Effluents

#### SAMPLING:

Quantity Required

: 10 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Ortho-phosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.5 at the full scale level.

Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

# **INSTRUMENTATION:**

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using IR sensitive phototube. Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; standard every 20 samples

# PHOSPHORUS, REACTIVE ortho-PHOSPHATE

#### QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90

Lab: Colourimetry

Analytical Range: - to 10.0 mg/L as P

# CALIBRATION CONTROL:

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
	******	*********			************
A :	153	8.0	7.998	-0.002	0.068
B :	153	4.0	4.003	0.003	0.035
A+B:	153	12.0	12.002	0.002	0.080
A-B:	153	4.0	3.994	-0.006	0.072
C :	153	4.0	4.003	0.003	0.035
D :	153	0.8	0.812	0.012	0.016
C+D:	153	4.8	4.816	0.016	0.042
C-D:	153	3.2	3.191	-0.009	0.035

s.d.(AB) S(between runs): 0.054

Sw(within run): 0.051 S/Sw: 1.06

s.d.(CD) S(between runs): 0.027

Sw(within run): 0.025 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

11.55 - 12.45 for A+B 3.70 - 4.30 for A-B 4.62 - 4.98 for C+D 3.08 - 3.32 for C-D

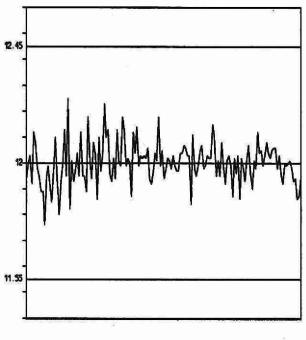
#### **DUPLICATES:**

Number of Data Pairs	C	Samp oncn S		Mean(2) s.d.	Coefficient of var.(%)
******			****		************
156	0.00		0.04	0.0105	77.5
42	0.04		0.10	0.0264	45.7
38	0.10		0.20	0.0364	23.8
55	0.20	-	1.00	0.0407	14.8
28	1.00	•	10.00	0.1162	4.3
319	(	Overal	1	0.0238	

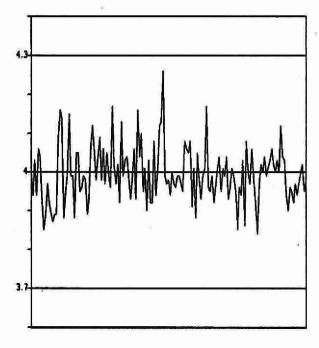
	Number of Data	Data Mean	Standard(1) Deviation
		****	
Long Term Blank	153	-0.0032	0.008

# PHOSPHORUS, REACTIVE ortho-PHOSPHATE (mg/L as P)

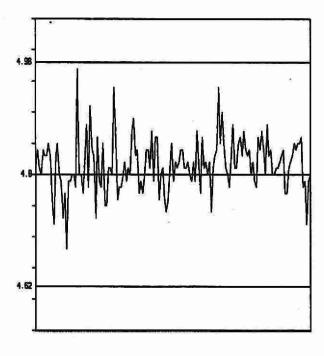
QUALITY CONTROL DATA FROM 02/01/90 TO 21/12/90



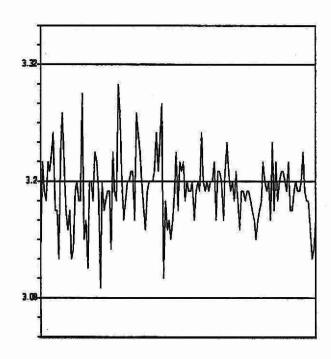
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# *** PHOSPHORUS, TOTAL ***

#### **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: 01/04/79

LIS Test Name Code Work Station Code : PPUT : RTNP Units
Unit Code
Supervisor

: mg/L as P : 064815 : M. Rawlings

Method Code Sample Type/Matrix : 504AC2 Supervisor : Rivers, Lakes, Precipitation, Soil Extracts, Effluents

#### SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digesters kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the full scale level. Total Kjeldahl nitrogen is determined simultaneously.

#### INSTRUMENTATION:

Three Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.002

T value: 0.01

# **CALIBRATION:**

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Recovery Drift : 3 digested BL plus 3 digested standards in duplicate, e.g. R1

: BL every 10 samples; undigested standard every 20 samples

#### NOTES:

The two occurrences of low values for the C-D variate were shown to be due to contamination of the C standard. The data was reported normally on confirmation of the contamination.

# PHOSPHORUS, TOTAL

# QUALITY CONTROL DATA FROM 03/01/90 TO 19/12/90

Lab: Colourimetry

Analytical Range: - to 0.20 mg/L as P

#### CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*********		*********	***********	
<b>A</b> :	174	0.16	0.1595	-0.0005	0.0021
<b>B</b> :	174	0.08	0.0794	-0.0006	0.0009
A+B:	174	0.24	0.2389	-0.0011	0.0027
A-B:	174	0.08	0.0801	0.0001	0.0017
C :	174	0.08	0.0794	-0.0006	0.0009
<b>D</b> :	174	0.016	0.0161	0.0001	0.0006
C+D:	174	0.096	0.0956	-0.0004	0.0012
C-D:	174	0.064	0.0633	-0.0007	0.0010

s.d.(AB) S(between runs): 0.002

Sw(within run): 0.001 S/Sw: 1.3

s.d.(CD) S(between runs): 0.0008

Sw(within run): 0.0007 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.231		0.249	for	A+B
0.074		0.086	for	A-B
0.092	•	0.100	for	C+D
0.0616	2€	0.0664	for	C-D

#### RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard(1) Deviation
R1:	174	0.14	0.134	0.0138
R2: R3:	174 174	0.084 0.028	0.081 0.027	0.0055 0.0033

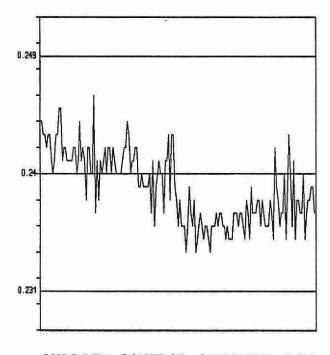
# **DUPLICATES:**

Number of Data Pairs	C	Sample onch Sp		Mean(2) s.d.	Coefficient of var.(%)
*****	*****		*****		***********
300	0.000	*	0.020	0.0019	27.8
106	0.020	•	0.050	0.0037	18.6
71	0.050	<b>W</b> .	0.200	0.0043	15.6
477	C	verall		0.0025	

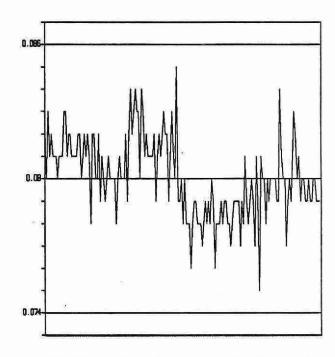
	of Data	Mean Mean	Standard(1) Deviation
•		********	-4
Long Term Blank Digested Blank	174 174	-0.0001 0.0019	0.0005 0.0021

# PHOSPHORUS, TOTAL (mg/L as P)

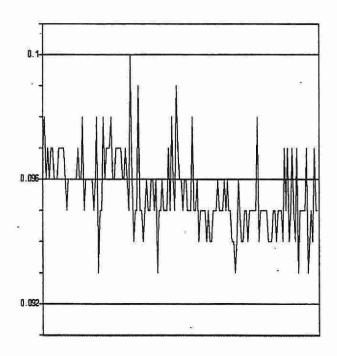
# QUALITY CONTROL DATA FROM 03/01/90 TO 19/12/90



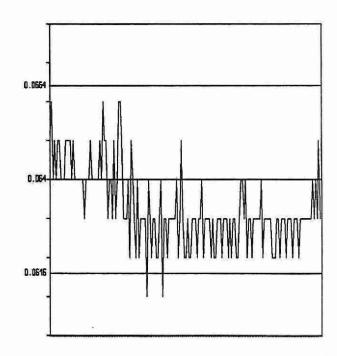
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# *** PHOSPHORUS, TOTAL ***

# **IDENTIFICATION:**

Laboratory

: Colourimetry

Method Introduced

: 01/04/79 : mg/L as P

LIS Test Name Code Work Station Code : PPUT : STKNP : 504BC2

Unit Code Supervisor

Units

: 064815 : M. Rawlings

Method Code Sample Type/Matrix

: Sewage, Industrial Waste, Leachate, Domestic Waters, Effluents

### SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using three block digestors kept at 180°C, 210°C and 360°C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.8 at the full scale level. Total Kjeldahl Nitrogen is determined simultaneously.

#### **INSTRUMENTATION:**

3-Block digesters

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using an IR sensitive phototube. Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

#### CALIBRATION:

BL plus 7 standards

# CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Recovery Drift : 3 digested BL plus 3 digested standards in duplicate, e.g. R1

: BL every 10 samples; undigested standard every 20 samples

#### PHOSPHOROUS, TOTAL

#### QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90

Lab: Colourimetry

Analytical Range: - to 10.0 mg/L as P

#### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*******	***********	**********		**********
A :	200	8.0	8.033	0.033	0.0320
B :	200	4.0	4.013	0.013	0.0187
A+B:	200	12.0	12.046	0.046	0.0412
A-B:	200	4.0	4.020	0.020	0.0324
C :	200	4.0	4.013	0.013	0.0187
D:	200	0.8	0.7998	-0.0002	0.0104
C+D:	200	4.8	4.8126	0.0126	0.0256
C-D:	200	3.2	3.2130	0.0130	0.0161

s.d.(AB) S(between runs): 0.026 Sw(wi

Sw(within run): 0.023 ' S/Sw: 1.14

s.d.(CD) S(between runs): 0.015

Sw(within run): 0.011 S/Sw: 1.33

On any given day the calibration is accepted if the values obtained lie within the ranges:

11.55		12.45	for	A+B
3.70		4.30	for	A-B
4.62	-	4.98	for	C+D
3.08	400	3.32	for	C-D

#### RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard(1) Deviation
				*************
R1:	200	7.0	6.88	0.142
R2:	200	4.2	4.15	0.071
R3:	200	1.4	1.39	0.059

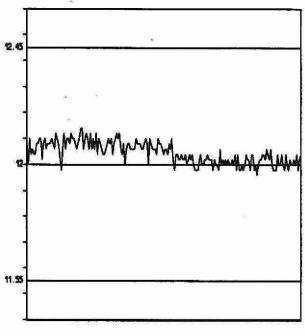
#### **DUPLICATES:**

Number of Data Pairs	C	Samp Concn S		Mean(2) s.d.	Coefficient of var.(%)
371	0.00		0.50	0.0164	9.5
64	0.50	-	1.00	0.0254	5.6
26	1.00	-	5.00	0.0407	
15	5.00	-	10.00	0.1082	6.6 2.3
476	)	Overall		0.0507	

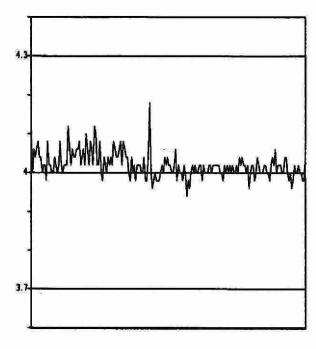
	Number of Data	Data Mean	Standard(1) Deviation
			************
Long Term Blank Digested Blank	200 200	0.0000 0.0010	0.005 0.007

# PHOSPHORUS, TOTAL (mg/L as P)

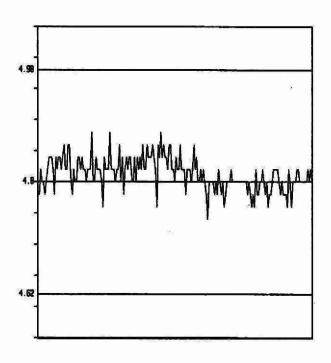
# QUALITY CONTROL DATA FROM 03/01/90 TO 24/12/90



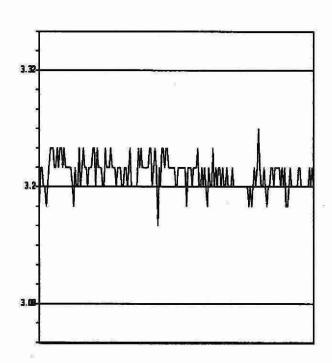
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

# *** PHOSPHORUS, TOTAL

#### **IDENTIFICATION:**

Laboratory

Method Code

LIS Test Name Code Work Station Code

: DOP : 5926C2

: PPUT1, PPUT2

: Dorset

Method Introduced Units

: 22/03/79 : ug/L as P : 063815

Unit Code Supervisor

: A. Neary

Sample Type/Matrix

: Streams, Lakes, Precipitation

#### SAMPLING:

Quantity Required

: 35 mL

Container

: Specially marked Pyrex culture tubes with Teflon-lined caps

#### ANALYTICAL PROCEDURE:

After withdrawal of excess volume, digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121°C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.3 at the full scale level

#### INSTRUMENTATION: •

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube. Two analytical ranges are obtained from the output of the colourimeter.

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.2

T value: 1

#### CALIBRATION:

BL plus 7 undigested standards

#### CONTROLS:

Calibration

: LTBL plus 4 undigested standards, e.g. QCA : 3 digested BL plus 3 digested standards, e.g. R1

Recovery Drift

: BL every 10 samples and BL plus 1 undigested standard every 20 samples

#### NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

The high bias encountered in (A+B) and (A-B) is currently under investigation.

#### PHOSPHORUS, TOTAL

#### QUALITY CONTROL DATA FROM 10/01/90 TO 21/12/90

Lab: Dorset

Analytical Range: - to 100.0 ug/L as P

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
				********	************
A :	67	75.0	78.06	3.06	2.04
B :	67	25.0	26.45	1.45	2.03
A+B:	67	100.0	104.52	4.52	3.31
A-B:	67	50.0	51.61	1.61	2.38
C :	67	7.5	7.66	0.11	1.85
D:	67	2.5	2.23	-0.27	0.50
C+D:	67	10.0	9.89	-0.11	1.21
C-D:	67	5.0	5.41	0.41	0.64

s.d.(AB) S(between runs): 2.1

Sw(within run): 1.7 S/Sw: 1.2

s.d.(AB) S(between runs): 0.61

Sw(within run): 0.45 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

90.5	<b>A</b> 1	109.5	for	A+B
43.0	<b>(2)</b>	57.0	for	A-B
7.4	(m)	12.6	for	C+D
3.0	187	7.0	for	C-D

#### RECOVERIES:

	Number of Data	.22()	Expected Concn	Av. Concn Measured	Standard(1) Deviation
			**********	**********	********
R1:	67		70.0	74.24	2.74
R2:	67		14.0	14.78	1.33
R3:	67	70	7.0	7.22	0.76

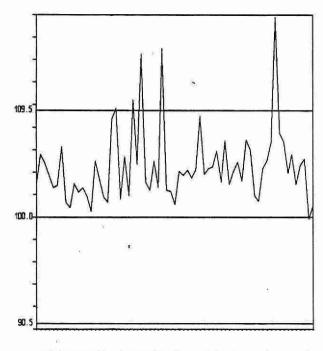
### **DUPLICATES:**

Number of Data Pairs		Sampl Concn S		Mean(2) s.d.	Coefficient of var.(%)
********	Lea		******	*******	
60	0.0	<b>5€</b> .	5.0	0.341	13.2
56	5.0	h.	10.0	0.375	9.7
46	10.0		20.0	0.617	7.6
38	20.0		100.0	1.293	4.4
200		Overall		0.509	

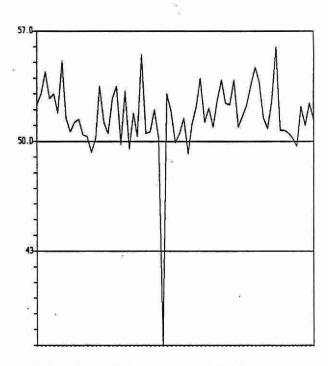
	Number of Data	Data Mean	Standard(1) Deviation	
	*********			
Long Term Blank Digested Blank	67 67	0.8 1.8	0.526 1.040	

# PHOSPHORUS, TOTAL (ug/L as P)

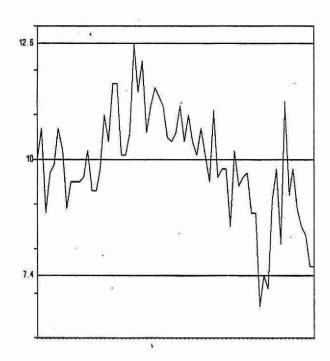
QUALITY CONTROL DATA FROM 10/01/90 TO 21/12/90



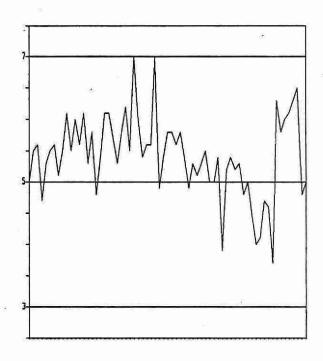
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

### *** POTASSIUM ***

# **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 18/05/79

Lis Test Name Code Work Station Code : KKUR : PRAA400 : 002EA1 Units
Unit Code
Supervisor

: mg/L as K : 064819 : M. Young

Method Code Sample Type/Matrix

: Precipitation, Throughfall, Filter extracts

#### SAMPLING:

Quantity Required

: 5 mL

Container

: Plastic

#### **ANALYTICAL PROCEDURE:**

Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

#### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.005

T value: 0.025

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL, reslope standard every 10 samples.

# **POTASSIUM**

# QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90

Lab: Atomic Absorption

Analytical Range: - to 1.00 mg/L as K

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
¥.		~~~~~~~	**********		************
A:	79	0.60	0.599	-0.001	0.0059
B :	79	0.10	0.103	0.003	0.0029
A+B:	79	0.70	0.702	0.002	0.0075
A-B:	79	0.50	0.496	-0.004	0.0055

s.d.(AB) S(between runs): 0.005 Sw(within run): 0.004 S/Sw:1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 0.75 for A+B 0.47 0.53 for A-B

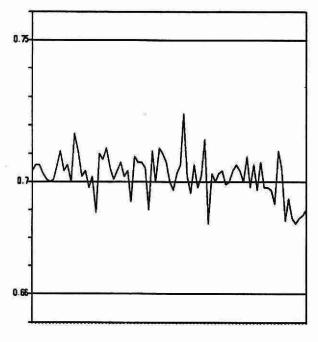
# **DUPLICATES:**

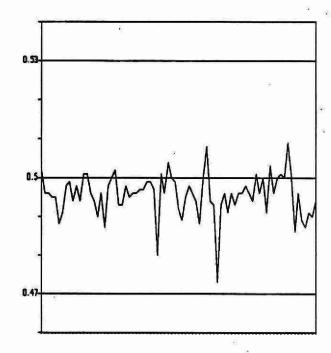
Number of Data Pairs		Samp onen S		Mean(2) s.d.	Coefficient of var.(%)
	****				
183	0.00	•	0.10	0.0016	7.6
24	0.10	-	0.20	0.0036	10.6
8	0.20	-	0.50	0.0054	3.6
9	0.50	-	1.00	0.0079	5.2
224		)veral	lu.	0.0020	And some

4	Number of Data		Standard(1) Deviation
	*******	*******	
Long Term Blank	79	0.00099	0.0033

# POTASSIUM (mg/L as K)

# QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

#### *** POTASSIUM ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 20/07/88

LIS Test Name Code Work Station Code

: KKUR : PRAAS Units Unit Code : mg/L as K : 064819

Method Code

: 002EA1

Supervisor

: M. Young

Sample Type/Matrix

: Rivers, Lakes

#### SAMPLING:

Quantity Required Container

: 5 mL

: Plastic

#### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

#### **INSTRUMENTATION:**

Automated modular atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g., QCA

Drift

: BL, reslope standard every 10 samples.

#### **POTASSIUM**

# QUALITY CONTROL DATA FROM 04/01/90 TO 21/12/90

Lab: Atomic Absorption

Analytical Range: - to 1.0 mg/L as K

# CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
				***********	
<b>A</b> :	95	0.80	0.797	-0.003	0.0072
B :	95	0.20	0.199	-0.001	0.0045
A+B:	95	1.00	0.996	-0.004	0.0099
A-B:	95	0.60	0.598	-0.002	0.0067
C:	95	0.20	0.199	-0.001	0.0045
D:	95	0.05	0.0501	0.0001	0.0030
C+D:	95	0.25	0.249	-0.001	0.0052
C-D:	95	0.15	0.149	-0.001	0.0055

s.d.(AB) S(between runs): 0.006

Sw(within run): 0.005 S/Sw: 1.3

s.d.(CD) S(between runs): 0.0038

Sw(within run): 0.0039 S/Sw: 0.98

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.955 1.045 for A+B 0.570 0.630 for A-B 0.295 for 0.205 C+D 0.120 C-D 0.180 for

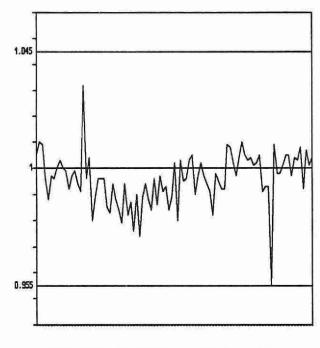
#### **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
******		*******		
56	0.00	- 0.20	0.0039	5.4
126	0.20	- 0.50	0.0068	2.1
81	0.50	- 1.00	0.0089	1.5
263	Ove	erall	0.0070	

	Number of Data	Data Mean	Standard(1) Deviation
			**********
Long Term Blank	94	-0.00006	0.0064

# POTASSIUM (mg/L as K)

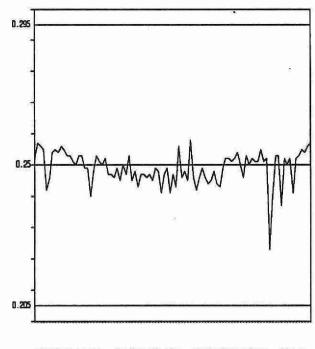
# QUALITY CONTROL DATA FROM 04/01/90 TO 21/12/90



0.63

QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B



0.15

QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

# *** POTASSIUM ***

#### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 18/05/79

LIS Test Name Code

: KKUR

Units

: ug/Filter as K

Work Station Code Method Code

: PRLOVAA : 004BA3

Unit Code Supervisor : 361819 : M. Young

Sample Type/Matrix

: W40 filters from LoVol filter packs

#### SAMPLING:

Quantity Required

: 1 filter

Container

: 50 mL Polyethylene tube

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### **ANALYTICAL PROCEDURE:**

Samples are analyzed by AAS at workstation PRAA400, at 766.5 nm using an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train. Results are converted to ug/filter as K.

Approximate absorbance: 0.5 at the full scale level.

#### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.25

T value: 1.25

# **CALIBRATION:**

BL plus 5 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL, reslope standard 10 samples

#### NOTES:

W and T values are those of the PRAA400 workstation multiplied by 50 to yield ug/filter.

#### *** POTASSIUM ***

#### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 01/04/74

Lis Test Name Code Work Station Code : KKUR : RMAAS Units
Unit Code

: mg/L as K

Method Code

: 0905A1

Supervisor

: 064819 : M. Young

Sample Type/Matrix

: Rivers, Lakes, Soil Extracts, Stemflow.

#### SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

#### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.923 at the full scale value.

#### INSTRUMENTATION:

Automated flow injection atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 11 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards e.g. QCA

Drift

: BL every 10 samples; 2 standards every 20 samples

#### **POTASSIUM**

# QUALITY CONTROL DATA FROM 09/01/90 TO 17/10/90

Lab: Atomic Absorption

Analytical Range: - to 5.00 mg/L as K

#### CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			*********		*******
<b>A</b> :	75	4.00	3.971	-0.029	0.0576
B :	75	1.00	0.996	-0.004	0.0171
A+B:	75	5.00	4.966	-0.034	0.0660
A-B:	75	3.00	2,975	-0.025	0.0535
C :	75	1.00	0.996	-0.004	0.0171
D:	75	0.25	0.249	-0.001	0.0089
C+D:	75	1.25	1.245	-0.005	0.0211
C-D:	75	0.75	0.746	-0.004	0.0172

s.d.(AB) S(between runs): 0.042

Sw(within run): 0.038 S/Sw:1.1

s.d.(CD) S(between runs): 0.014

Sw(within run): 0.012 S/Sw:1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.770 - 5.230 for A+B 2.850 - 3.150 for A-B 1.175 - 1.325 for C+D 0.700 - 0.800 for C-D

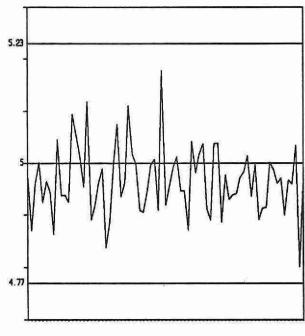
#### **DUPLICATES:**

Number of Data Pairs	Sample Concn Span				Coefficient of var.(%)
******					*************
28	0.00		0.25	0.0035	2.8
44	0.25	-	1.00	0.0162	3.2
99	1.00	-	2.50	0.0275	14.2
37	2.50		5.00	0.0798	11.2
208	(	Overal	1	0.0275	RAPSET I

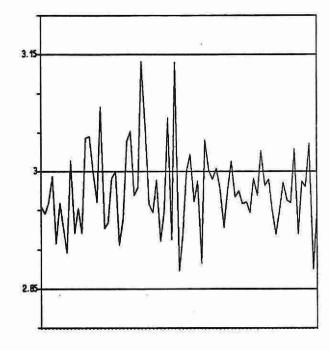
	Number of Data	Data Mean	Standard(1) Deviation
		**********	
Long Term Blank	75	-0.0028	0.0325

# POTASSIUM (mg/L as K)

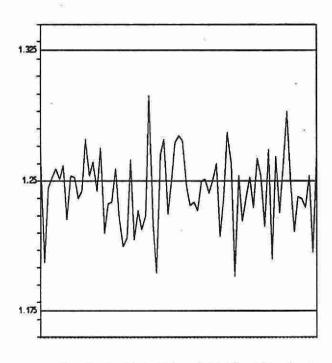
# QUALITY CONTROL DATA FROM 09/01/90 TO 17/12/90



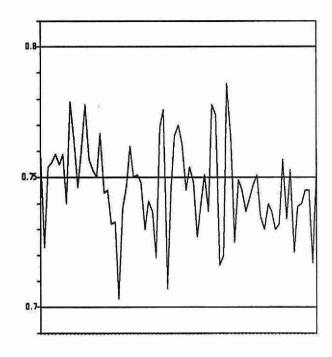
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

#### *** POTASSIUM ***

### **IDENTIFICATION:**

Laboratory
Lis Test Name Code

: Atomic Absorption

Method Introduced Units

: 08/04/86 : mg/L as K

Work Station Code
Method Code

: KKUR : WAAS : 002EA1

Unit Code Supervisor : 064819 : M. Young

Sample Type/Matrix

: Domestic Waters, Leachates, Effluents, Sewage, Industrial wastes

# **SAMPLING:**

Quantity Required

: 6 mL

Container

: Glass or Plastic

#### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.16 at full scale level.

#### **INSTRUMENTATION:**

Automated flow injection atomic absorption spetrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

#### CALIBRATION:

BL plus 11 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards e.g. QCA

Drift

: BL every 10 standards; 2 standards every 20 samples

#### **POTASSIUM**

# QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90

Lab: Atomic Absorption

Analytical Range: - to 25.0 mg/L as K

#### **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
			***************************************	*******	
A :	74	20.00	20.003	0.003	0.3560
B:	74	5.00	4.97	-0.03	0.1443
A+B:	74	25.00	24.97	-0.03	0.4049
A-B:	74	15.00	15.04	0.04	0.3621
C:	74	5.00	4.97	-0.03	0.1443
D:	74	1.25	1.247	-0.003	0.0824
C+D:	74	6.25	6.21	-0.04	0.1807
C-D:	74	3.75	3.72	-0.03	0.1502

s.d.(AB) S(between runs): 0.27

Sw(within run): 0.26 S/Sw: 1.1

s.d.(CD) S(between runs): 0.12

Sw(within run): 0.11 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.8		26.2	for	A+B
14.2		15.8	for	A-B
5.65	•	6.85	for	C+D
3 35		4 15	for	C-D

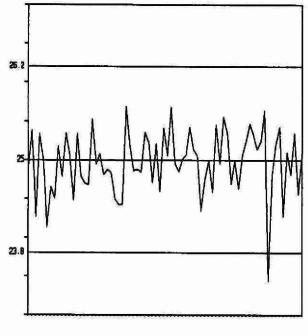
#### **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
				**********	************
38	0.00	,# <b>E</b>	1.25	0.0414	5.5
70	1.25		2.50	0.0554	4.3
32	2.50	-	5.00	0.0774	4.4
17	5.00	-	25.00	0.2685	2.3
157	(	Overal	1	0.0658	

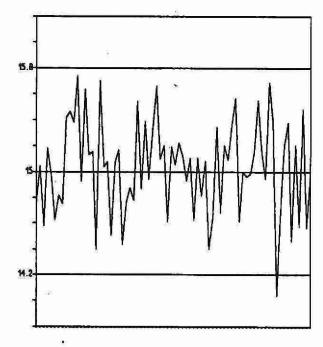
	Number of Data	Data Mean	Standari(1) Deviation
	**********		
Long Term Blank	52	-0.0772	0.1647

# POTASSIUM (mg/L as K)

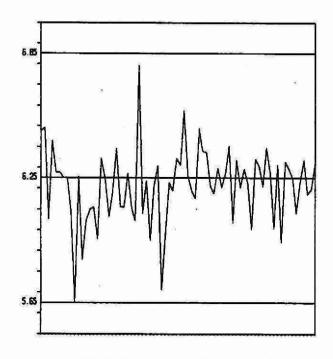
# QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90



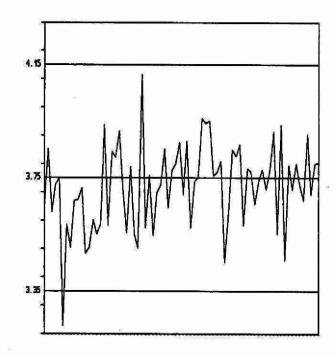
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

### *** POTASSIUM, EXCHANGEABLE CATION ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80

LIS Test Name Code Work Station Code

: KKESC : DOCATION Units Unit Code Supervisor : meg/100 g : 355000 : A. Neary

Method Code Sample Type/Matrix : 306AA1

: Soil

#### SAMPLING:

Quantity Required

: 6 g dry

Container

: Glass

#### SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

#### ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for K by AAS at 766.5 nm with an air-acetylene flame. Approximate absorbance: 0.3 at the full scale level. N.B. Aluminum, calcium, and magnesium are determined on the same extract.

#### INSTRUMENTATION:

- -Varian AA1275 with programmable sampler changer and Gilson Minipuls II pump
- -Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

Three soil samples representing different soil types; 2 OC solutions at 25% and 75% of

full scale; 2 method blanks; round robin ECSS samples (run occasionally)

Drift

:BBL plus 1 standard (100% F.S.) every 10 samples

#### NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown - average value used.

# POTASSIUM, EXCHANGEABLE CATION

# QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90

Lab: Dorset Soils

Analytical Range: - to 0.75 meq/100 g

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
					***********
<b>A</b> :	17	0.56	0.566	0.006	0.0132
В:	17	0.19	0.187	-0.003	0.0098
A+B:	17	0.75	0.753	0.003	0.0215
<b>A-B</b> :	17	0.38	0.379	-0.001	0.0090

s.d.(AB) S(between runs): 0.012 Sw(within run): 0.006 S/Sw: 1.83

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 0.66 0.84 for A+B 0.32 0.44 for A-B

# **RECOVERIES:**

	Number, of Data	Av. Concn Measured	Standard(1) Deviation
	**********		
R1:	17	0.388	0.0175
R2:	17	0.156	0.0413
R3:	17	0.125	0.0112

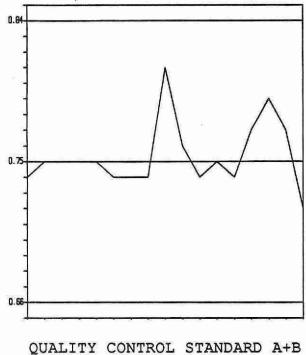
#### **DUPLICATES:**

Number of Data Pairs	Sample Conen Span		Mean(2) s.d.	Coefficient of var.(%)	
*********				******	**********
17	0.00		0.15	0.0126	9.0
19	0.15		0.25	0.0144	6.5
14	0.25		0.75	0.0146	5.2
50	(	Overall		0.0136	

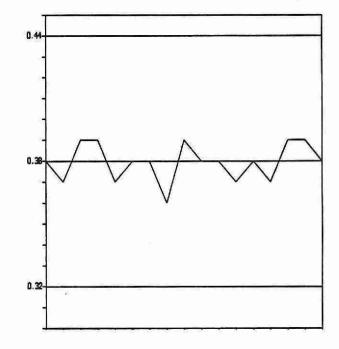
	Number of Data	Data Mean	Standard(1) Deviation
		**********	
Digested Blank	17	-0.0006	0.0024

# POTASSIUM, EXCHANGEABLE CATION (meq/100 g as K)

QUALITY CONTROL DATA FROM 02/01/90 TO 01/11/90







QUALITY CONTROL STANDARD A-B

# *** SAND ***

#### **IDENTIFICATION:**

Laboratory

LIS Test Name Code Work Station Code Method Code

Sample Type/Matrix

: Dorset Soils

: SAND : DOPARTSZ

: AM1002 : Soil

Method Introduced

Units Unit Code Supervisor : 01/06/80

: % by weight : 070000 : A. Neary

# SAMPLING:

Quantity Required

: 20 g dry

Container

: Glass or plastic

# SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved <2 mm.

### ANALYTICAL PROCEDURE:

To prevent flocculation a portion of sample, pretreated for organic matter and carbonate removal, is dispersed in a sodium hexametaphosphate solution. The sand fraction (>53 um) is removed by wet sieving; the silt and clay fraction is dispersed in a sedimentation cylinder. The percentage of sand in the sample is determined by weighing the dried sieved fraction and expressing that as a percentage by weight of the total (sand, silt and clay) recovered.

#### INSTRUMENTATION:

-Sartorius 4 place digital balance (Handy)

-Balance accurate to 0.0001 g

### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 1

T value: 5

# CALIBRATION:

Balance zero

#### CONTROLS:

Recovery

: 2 long term soil samples representing different soil types plus round robin ECSS

samples (run occasionally).

# **SAND**

# QUALITY CONTROL DATA FROM 01/06/90 TO 22/11/90

Lab: Dorset Soils

Analytical Range: - to 100 % by wt.

# **RECOVERIES:**

	Number	Av. Concn	Standard(1)
	of Data	Measured	Deviation
		*************	
R1:	9	4.556	0.527
R2:	9	55.778	1.202

# **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		ple	Mean(2) s.d.	Coefficient of var.(%)
4	0.0	-	25.0	0.3535	2.3
2	25.0	1	50.0	N.A	N.A
7	50.0		100.0	2.601	4.4
12	(	Over	all	1.645	

# *** SILICON, ACID AMMONIUM OXALATE EXTRACTABLE ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 1986

LIS Test Name Code Work Station Code : SIEOX : DOMETOX

Units Unit Code : % by wt as Si : 070814

Method Code Sample Type/Matrix : 302AA5 : Soil Supervisor : A. Neary

# SAMPLING:

Quantity Required

: 1 g

Container

: Glass or plastic

### SAMPLE PREPARATION:

Samples are air-dried, disaggregated and sieved to less than 2 mm. A subsample is ground to <500 um (35 mesh).

# **ANALYTICAL PROCEDURE:**

Samples are weighed into disposable tubes. 10 mL of acid ammonium oxalate extractant is added and the tubes are capped and shaken for 4 hours in the dark. Samples are then centrifuged and the analysis is performed on the supernatant.

#### INSTRUMENTATION:

Varian AA 1275

#### REPORTING:

Maximum Significant Figures: 2

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

:Three long term soil samples representing different soil types, 2 method blanks, 2

QC solutions at 25% and 75% of scale, round robin ECSS samples.

Drift

:BL plus 1 standard (100% F.S.) every 10 samples.

# SILICON, ACID AMMONIUM OXALATE EXTRACTABLE

# QUALITY CONTROL DATA FROM 30/03/90 TO 14/12/90

Lab: Dorset Soils

Analytical Range: - to 0.25 % wt. Si

# **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
		**********	************		
A :	4	0.180	0.185	0.005	0.010
B :	4	0.060	0.060	0.000	0.014
A+B:	4	0.240	0.245	0.005	0.021
A-B:	4	0.120	0.125	0.005	0.013

s.d.(AB) S(between runs): 0.122 Sw(within run): 0.009 S/Sw: 1.34

# **RECOVERIES:**

	Number of Data	Av. Concn Measured	Standard(1) Deviation
	******	*******	
R1:	4	0.057	0.009
R2:	4	0.162	0.112
R3:	4	0.140	0.095

# **DUPLICATES:**

Number of	Sample		Mean(2)	Coefficient	
Data Pairs	Concn Span		s.d.	of var.(%)	
	***************************************				
6	0.0		0.15	0.0029	3.8

	Number	Data	Standard(1)
	of Data	Mean	Deviation
Method Blank	4	0.000	0.000

# *** SILICON, REACTIVE SILICATES

# **IDENTIFICATION:**

Laboratory

LIS Test Name Code Work Station Code

Method Code

: SIO3UR : ROM

: Colourimetry

: 001BC1

Method Introduced

: 01/02/75 Units : mg/L as Si Unit Code : 064814

Supervisor

: M. Rawlings

Sample Type/Matrix

: Rivers, Lakes, Precipitation, Soil Extracts, Effluents Domestic Water Supplies,

Leachates

# SAMPLING:

Quantity Required

: 10 mL : Plastic

Container

### ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the full scale level.

Dissolved inorganic and dissolved organic carbon are determined simultaneously.

### **INSTRUMENTATION:**

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 660 nm. Data capture, reduction, and processing via a multi-stage microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

#### CALIBRATION:

BL plus 7 standards

#### **CONTROLS:**

Calibration

: LTBL plus 3 standards, e.g. OCA

Drift

: BL every 10 samples; standards every 20 samples

#### NOTES:

Calibration standard is a hydrate: Na₂SiO₃.9H₂O.

# SILICON, REACTIVE SILICATES

# QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90

Lab: Colourimetry

Analytical Range: - to 10.0 mg/L as Si

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		********	*********		***********
A :	198	8.0	7.93	-0.07	0.065
B :	198	2.0	1.99	-0.01	0.031
A+B:	198	10.0	9.92	-0.08	0.092
A-B:	198	6.0	5.95	-0.05	0.084
C :	198	2.0	1.99	-0.01	0.058
D :	198	0.5	0.51	0.01	0.031
C+D:	198	2.5	2.49	-0.01	0.050
C-D:	198	1.5	1.48	-0.02	0.018

s.d.(AB) S(between runs): 0.05

Sw(within run): 0.04 S/Sw: 1.24

s.d.(CD) S(between runs): 0.03

Sw(within run): 0.01 S/Sw: 2.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.70 - 10.30 for A+B 5.80 - 6.20 for A-B 2.30 - 2.70 for C+D 1.38 - 1.62 for C-D

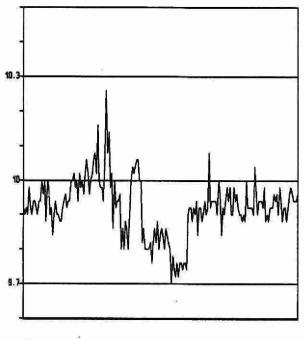
# **DUPLICATES:**

Number of Data Pairs	C	Samp oncn S		Mean(2) s.d.	Coefficient of var.(%)
193	0.00	-	1.00	0.016	3.9
81	1.00	:=:	2.00	0.015	3.0
180	2.00	100	5.00	0.020	0.7
92	5.00	-	10.00	0.041	0.7
546		Overal	1	0.017	25 740

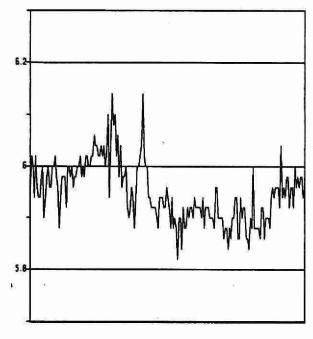
	Number of Data	Data Mean	Standard(1) Deviation
	*****		
Long Term Blank	198	-0.008	0.016

# SILICON, REACTIVE SILICATES (mg/L as Si)

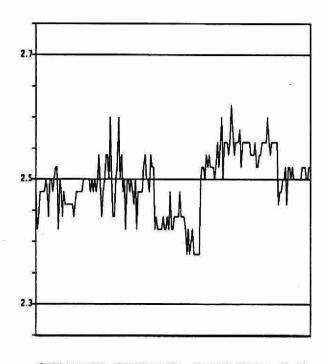
QUALITY CONTROL DATA FROM 04/01/90 TO 20/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

- 320 -

#### *** SILT ***

#### **IDENTIFICATION:**

Laboratory

: Dorset Soils

Method Introduced

: 01/06/80 : % by weight

LIS Test Name Code Work Station Code : SILT : DOPARTSZ Units Unit Code

Work Station Cod Method Code

: AM1002

Supervisor

: 070000 : A. Neary

Sample Type/Matrix

: Soil

# SAMPLING:

Quantity Required

: 20 g dry

Container

: Glass or plastic

#### **SAMPLE PREPARATION:**

Samples are air dried, disaggregated and sieved to <2 mm.

#### ANALYTICAL PROCEDURE:

To prevent flocculation a portion of sample, pretreated for organic matter and carbonate removal, is dispersed in a sodium hexametaphosphate solution. The sand fraction (>53 um) is removed by wet sieving; the silt and clay fraction is dispersed in a sedimentation cylinder. The percentage of silt in the sample is based on the settling velocities of spherical particles by the application of Stokes Law.

#### INSTRUMENTATION:

-Sartorius 4 place digital balance (Handy)

-Balance accurate to 0.0001 g

### REPORTING:

Maximum Significant Figures: 3

Calculated W value: 1

T value: 5

#### CALIBRATION:

Balance zero

#### CONTROLS:

Recovery

: 2 long term soil samples representing different soil types plus a round robin ECSS sample

(run occasionally).

# SILT

# QUALITY CONTROL DATA FROM 01/06/90 TO 22/11/90

Lab: Dorset Soils

Analytical Range: - to 100 % by wt.

# **RECOVERIES:**

	Number	Av. Concn	Standard(1)
	of Data	Measured	Deviation
			**********
R1:	9	43.11	1.76
R2:	10	41.20	1.75

# **DUPLICATES:**

Number of	Sample			Mean(2)	Coefficient
Data Pairs			Span	s.d.	of var.(%)
4	0.0	<b>=</b> 0	20.0	1.389	9.3
3	20.0	=	50.0	2.121	5.0
4	50.0	-	100.0	1.500	2.0
11		)vera	all	1.657	

# *** SODIUM ***

### **IDENTIFICATION:**

Laboratory

: Atomic Absorption : NAUR

Method Introduced

: 18/05/79

Lis Test Name Code Work Station Code

: PRAA400

Units Unit Code Supervisor : mg/L as Na : 064811 : M. Young

Method Code Sample Type/Matrix : 001EA1

: Precipitation, Throughfall, Filter extracts

### SAMPLING:

Quantity Required

: 5 mL : Plastic

Container

#### .

# ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

#### INSTRUMENTATION:

Automated modular atomic absorption spectrophotometer (AAS) system.

# REPORTING:

Maximum Significant Figures: 3

Current W value: 0.005

T value: 0.025

#### CALIBRATION:

BL plus 5 standards

# CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL, reslope standard every 10 samples.

# SODIUM

# QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90

Lab: Colourimetry

Analytical Range: - to 1.00 mg/L as Na

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	78	0.60	0.599	-0.001	0.0063
<b>B</b> :	78	0.10	0.103	0.003	0.0038
A+B:	78	0.70	0.702	0.002	0.0082
A-B:	78	0.50	0.496	-0.004	0.0064

s.d.(AB) Sw(within run): 0.0052

S(between runs): 0.0045 S/Sw: 1.15

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 - 0.75 for A+B 0.47 - 0.53 for A-B

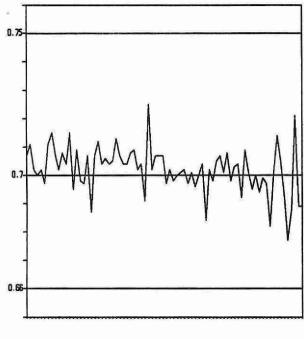
# **DUPLICATES:**

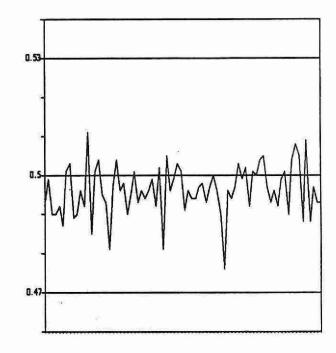
Number of Data Pairs		Samp onen S		Mean(2) s.d.	Coefficient of var.(%)
163	0.00	<b>S</b> 7	0.10	0.0014	29.7
31	0.10	-	0.20	0.0030	2.7
14	0.20		0.50	0.0053	1.9
8	0.50		1.00	0.0120	2.0
216	C	)veral	1	0.0019	

	Number of Data	Data Mean	Standard(1) Deviation
9			***********
Long Term Blank	78	-0.00065	0.0065

# SODIUM (mg/L as Na)

# QUALITY CONTROL DATA FROM 05/01/90 TO 28/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CON.

CONTROL LIMIT

### *** SODIUM ***

# **IDENTIFICATION:**

Laboratory

Method Code

: Atomic Absorption

Method Introduced

: 20/07/88

LIS Test Name Code Work Station Code : NAUR : PRAAS : 001EA1 Units
Unit Code
Supervisor

: mg/L as Na : 064811 : M. Young

Sample Type/Matrix

: Rivers, Lakes

# SAMPLING:

Quantity Required

: 5 mL : Plastic

Container

# ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level.

# **INSTRUMENTATION:**

Automated modular atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.01

T value: 0.05

#### CALIBRATION:

BL plus 5 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g., QCA

Drift

: BL, reslope standard every 10 samples.

# SODIUM

# QUALITY CONTROL DATA FROM 04/01/90 TO 21/12/90

Lab: Atomic Absorption

Analytical Range: - to 4.0 mg/L as Na

# **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
*	of Data	Concn	Measured	Bias	Deviation
	*********			********	
<b>A</b> :	96	3.2	3.207	0.007	0.0240
B :	96	0.8	0.808	0.008	0.0075
A+B:	96	4.0	4.015	0.015	0.0271
A-B:	96	2.4	2.3996	-0.0004	0.0231
C :	96	0.8	0.808	0.008	0.0075
D:	96	0.2	0.206	0.006	0.0073
C+D:	96	1.0	1.014	0.014	0.0104
C-D:	96	0.6	0.601	0.001	0.0106

s.d.(AB) S(between runs): 0.018

Sw(within run): 0.016 S/Sw:1.1

s.d.(CD) S(between runs): 0.0074

Sw(within run): 0.0075 S/Sw:1.0

On any given day the calibration is accepted if the values obtained lie within the ranges:

3.82		4.18	for	A+B
2.28	=	2.52	for	A-B
0.82	-	1.18	for	C+D
0.48		0.72	for	C-D

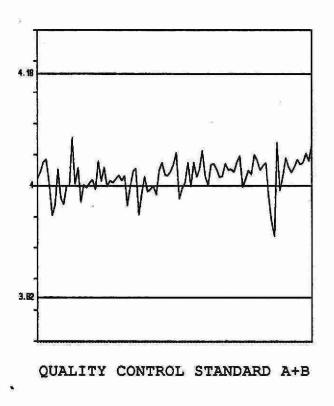
# **DUPLICATES:**

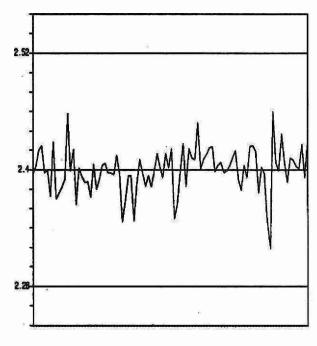
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
	*****			
41	0.00	- 0.60	0.0081	1.9
177	0.60	- 2.00	0.0184	2.6
34	2.00	4.00	0.0375	1.3
252	Ove	erall	0.0193	

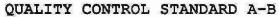
	Number	Data	Standard(1)
	of Data	Mean	Deviation
		************	**********
Long Term Blank	93	-0.0007	0.0072

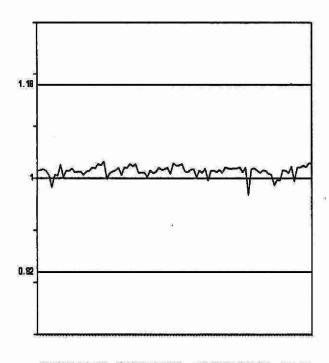
# SODIUM (mg/L as Na)

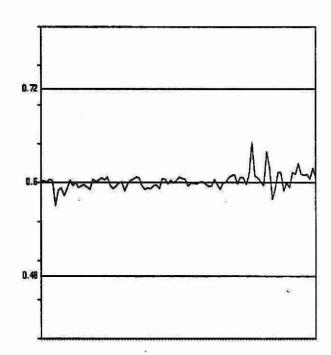
# QUALITY CONTROL DATA FROM 04/01/90 TO 21/12/90











QUALITY CONTROL STANDARD C+D

QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

#### *** SODIUM ***

# **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 18/05/79

LIS Test Name Code

: NAUR : PRLOVAA Units Unit Code : ug/Filter as Na

Work Station Code Method Code

: 004AA3

Supervisor

: 361811 : M. Young

Sample Type/Matrix

: W40 filters from LoVol filter packs

#### SAMPLING:

Quantity Required

: 1 filter

Container

: 50 mL Polyethylene tube

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at workstation PRAA400, at 589.0 nm using an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train. Results are converted to ug/filter as Na. Approximate absorbance: 0.5 at the full scale level.

### **INSTRUMENTATION:**

Automated modular atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.25

T value: 1.25

#### CALIBRATION:

BL plus 5 standards.

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: BL, reslope standard every 10 samples.

#### NOTES:

W and T values are those of the PRAA400 workstation multiplied by 50 to yield ug/filter.

### *** SODIUM ***

# **IDENTIFICATION:**

Laboratory Lis Test Name Code

: Atomic Absorption : NAUR

Method Introduced Units

: 01/04/74 : mg/L as Na

Work Station Code Method Code

: RMAAS : 0905A1

Unit Code Supervisor

: 064811 : M. Young

Sample Type/Matrix

: Rivers, Lakes, Soil Extracts, Stemflow.

# SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

# **ANALYTICAL PROCEDURE:**

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.16 at the full scale value.

# **INSTRUMENTATION:**

Automated flow injection atomic absorption spectrophotometer (AAS) system.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.02

T value: 0.1

# **CALIBRATION:**

BL plus 11 standards

# CONTROLS:

Calibration

: LTBL plus 3 standards e.g. OCA

Drift

: BL every 10 samples; 2 standards every 20 samples

# SODIUM

# QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90

Lab: Atomic Absorption

Analytical Range: - to 20.0 mg/L as Na

# **CALIBRATION CONTROL:**

	Number	Expected	Av. Concn	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
				*******	********
<b>A</b> :	93	16.0	15.903	-0.007	0.1872
B :	93	4.0	3.996	-0.004	0.0743
A+B:	93	20.0	19.900	-0.100	0.2229
A-B:	93	12.0	11.907	-0.003	0.1774
<b>C</b> :	93	4.0	3.996	-0.004	0.0743
D:	93	1.0	1.006	0.006	0.0382
C+D:	93	5.0	5.002	0.002	0.0868
C-D:	93	3.0	2.991	-0.009	0.0801

s.d.(AB) S(between runs): 0.14

Sw(within run): 0.12 S/Sw: 1.13

s.d.(CD) S(between runs): 0.059

Sw(within run): 0.057 S/Sw: 1.04

On any given day the calibration is accepted if the values obtained lie within the ranges:

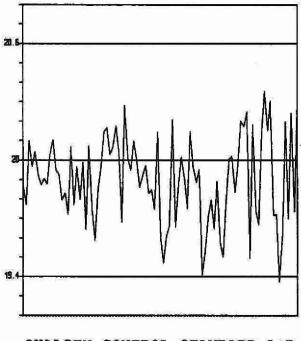
19.4		20.6	for	A+B
11.6	656	12.4	for	A-B
4.7		5.3	for	C+D
2.8	700	3.2	for	C-D

# **DUPLICATES:**

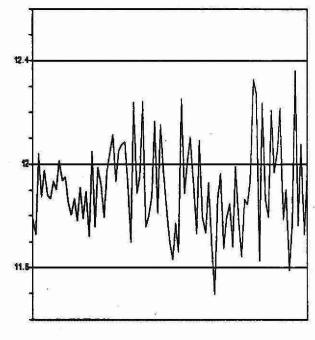
Number of Data Pairs	c	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
	****				
83	0.00	-	1.00	0.0212	5.2
21	1.00	•	2.00	0.0499	3.5
42	2.00		5.00	0.0652	1.9
52	5.00	#1 P	10.00	0.1162	1.5
35	10.00	-	20.00	0.2436	1.8
233	(	Overal	1	0.0725	

	Number of Data	Data Mean	Standard(1) Deviation
			*********
Long Term Blank	93	0.0033	0.0271

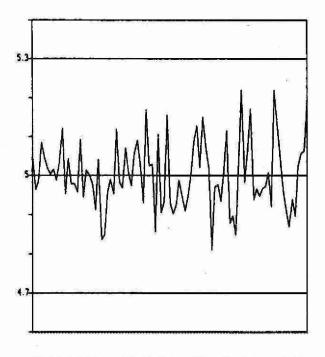
# QUALITY CONTROL DATA FROM 09/01/90 TO 21/12/90



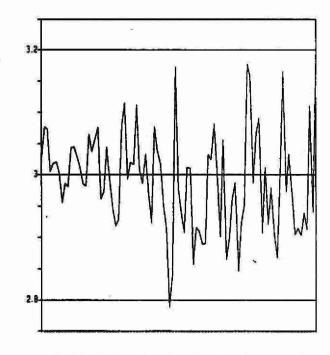
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

# *** SODIUM ***

### **IDENTIFICATION:**

Laboratory

: Atomic Absorption

Method Introduced

: 08/04/86 : mg/L as Na

Lis Test Name Code Work Station Code : NAUR : WAAS Units Unit Code

: 064811

Method Code

: 001EA1

Supervisor

: M. Young

Sample Type/Matrix

: Domestic Waters, Leachates, Effluents, Sewage, Industrial wastes

# SAMPLING:

Quantity Required

: 6 mL

Container

: Glass or Plastic

#### **ANALYTICAL PROCEDURE:**

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Cesium chloride is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.21 at the full scale level.

# **INSTRUMENTATION:**

Automated flow injection atomic absorption spectrophotometer (AAS) system.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.2

T value: 1

#### CALIBRATION:

BL plus 11 standards

#### CONTROLS:

Calibration

: LTBL plus 3 standards, e.g. QCA

Drift

: BL every 10 samples; 2 standards every 20 samples

# SODIUM

# **QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90**

Lab: Atomic Absorption

Analytical Range: - to 100.0 mg/L as Na

# CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*******	*********			************
<b>A</b> :	137	80.0	80.29	0.29	1.4895
B :	137	20.0	19.95	-0.05	0.5381
A+B:	137	100.0	100.24	0.24	1.7578
A-B:	137	60.0	60.34	0.34	1.3879
C:	137	20.0	19.95	-0.05	0.5381
D:	137	5.0	4.97	-0.03	0.2701
C+D:	137	25.0	24.92	-0.08	0.6894
C-D:	137	15.0	14.98	-0.02	0.4998

s.d.(AB) S(between run): 1.12

Sw(within run): 0.98 S/Sw: 1.1

s.d.(CD) S(between run): 0.43

Sw(within run): 0.35 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

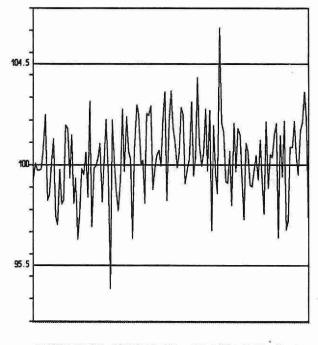
95.50 - 104.50 for A+B 57.00 - 63.00 for A-B 22.25 - 27.75 for C+D 13.50 - 16.50 for C-D

#### **DUPLICATES:**

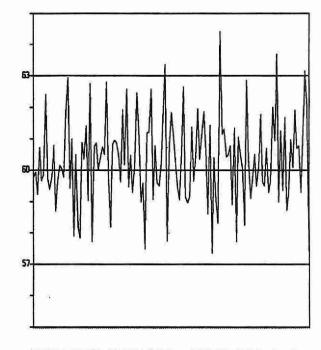
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
		******		00 0000 de 00 d	**************
114	0.0	•	10.0	0.1941	4.2
114	10.0	•	25.0	0.4371	2.9
57	25.0	-	50.0	0.7120	2.0
30	50.0	-	100.0	1,4919	2.1
315		Overal	1	0.4506	

i)c	Number	Data	Standard(1)
	of Data	Mean	Deviation
	*********		***********
Long Term Blank	125	-0.1441	0.3212

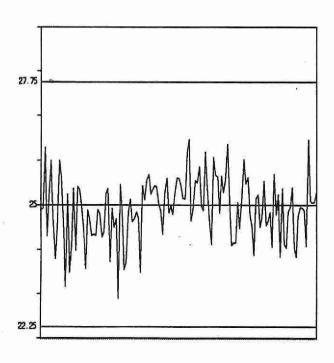
# QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90



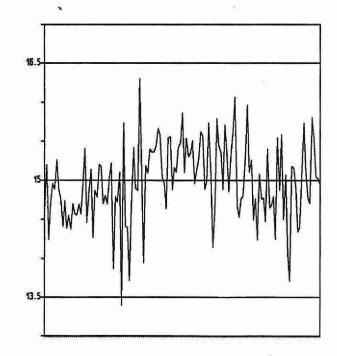
QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B



QUALITY CONTROL STANDARD C+D



QUALITY CONTROL STANDARD C-D

CONTROL LIMIT

# SOLIDS, DISSOLVED

#### **IDENTIFICATION:**

Laboratory

: Solids and BOD

Method Introduced

: Before '61

LIS Test Name Code Work Station Code

: RSF : SOLIDS Units Unit Code

: mg/L : 064000

Method Code

: 106AB4

' Supervisor

: P. Campbell

Sample Type/Matrix

: Sewage, Industrial Waste, Effluents, Domestic Waters, Surface Waters, Leachates

### SAMPLING:

Quantity Required

: 125 mL

Container

: Glass or plastic

# ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50 or 100 mL of filtrate is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. After reweighing the dish the dissolved solids content is calculated by subtracting the original dish mass from the dried dish mass. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

Balance (4/5 decimal places), drying oven, suction filtration apparatus, Teflon dishes Microcomputer system with appropriate software

### REPORTING:

Maximum Significant Figures: 3

Current W value: 2

T value: 10

#### CALIBRATION:

Balance zero

Internal calibration provided in the balance

#### CONTROLS:

Calibration

: 2 S class weights, e.g. QCA

: LTBL plus 2 standards, e.g. R1

Recovery Drift

: Balance zero is checked every 10 dishes.

#### NOTES:

Correction factor for dish tare weights is included in the calculation.

based on variations of a standard sealed vessel.

Two balances are used for all solids analyses. Currently tests on sewage are performed by a private laboratory. QC results reported here represent only tests performed at the Central Laboratory.

Duplicates in the lowest range class were generally too high to use to recalculate the W value.

# SOLIDS, DISSOLVED

# QUALITY CONTROL DATA FROM 26/03/90 TO 06/11/90

Lab: Solids and BOD

Analytical Range: - to 20000 mg/L

# CALIBRATION CONTROL:

	Number of Data	Expected Mass	Av. Mass Measured	Av. Bias	Standard(1) Deviation
		*********		***************************************	
A :	38	50.00	50.00013	0.00013	0.00015
B :	38	30.00	29.99971	-0.00029	0.00031
A+B:	38	80.00	79,99984	-0.00016	0.00039
A-B:	38	20.00	20.00041	0.00041	0.00028

s.d.(AB) S(between runs): 0.00024 Sw(within run): 0.00020 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

79.9994 - 80.0006 19.9994 - 20.0006

forA+B forA-B

#### **RECOVERIES:**

	Number	Expected	Av. Concn	Standard(1)
	of Data	Concn	Measured	Deviation
R1:	52	20000.0	20024.5	81.37
R2:	52	2000.0	1999.1	27.65
R3:	52	500.0	498.6	9.14

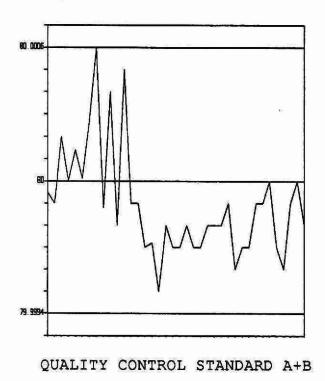
# **DUPLICATES:**

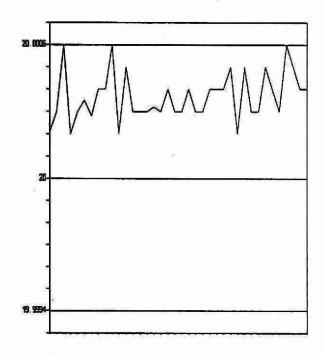
Number of Data Pairs	C	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
******			*******		************
34	0	_	250	9.67	8.3
37	250	-	500	16.78	4.0
49	500	•	1000	21.08	3.8
11	1000		3000	253.95	12.6
0	3000		20000	n.a.	n.a.
131	1	Overa	11	20.21	

	Number of Data	Data Mean	Standard(1) Deviation
		***********	*********
Blank	76	-0.9399	5.836

# SOLIDS, DISSOLVED (mg/L)

# QUALITY CONTROL DATA FROM 26/03/90 TO 06/11/90





QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

# *** SOLIDS, IGNITED ***

#### **IDENTIFICATION:**

Laboratory

: Solids and BOD

Method Introduced

: Before '81

LIS Test Name Code Work Station Code : RSFA,RSPA,RSTA : SOLIDS Units Unit Code

: mg/L or mg/Kg : 064000

Method Code

: 107AB4,207AB5,507AB4

Supervisor

: P. Campbell

Sample Type/Matrix

: Sewage, Industrial Waste, Effluents, Domestic Waters, Leachates

### SAMPLING:

Quantity Required

: 5-500 mL

Container

: Glass or plastic

### ANALYTICAL PROCEDURE:

The procedure for dissolved, particulate, or total solids is followed and the dried residue is ignited at 600°C for one hour in a muffle furnace. As soon as practical, the dish is transferred to a desiccator to cool. The ignited or ash mass is obtained as the difference between the final ignited mass and the original dish or filter mass. Similarly the volume used in the ignited calculations is the volume selected for the original dried solids measurement. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

### **INSTRUMENTATION:**

Balance (4/5 decimal places), muffle furnace, ceramic dishes, Petri dishes Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 2,0.5,2

T value: 10,2.5,10

#### CONTROLS:

Calibration

: 4 S class weights, e.g. OCA

Drift

: Balance zero is checked at least every 20 dishes.

#### NOTES:

In the order listed above, W and T values refer to the residual ash after ignition of the dried residual from dissolved, particulate, and total solids determinations.

Detection criteria estimates are unreliable due to limited data; samples requiring these tests are usually sewage sludges with high solids contents.

Currently tests on sewage are performed by a private laboratory. QC results reported here represent only tests performed at the Central Laboratory.

Insufficient tests were performed at the Central Laboratory to provide a data summary.

# *** SOLIDS, PARTICULATE ***

### **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code

Method Code

Sample Type/Matrix

: Solids and BOD

: RSP : SOLIDS

: 206AB5

Waters

Method Introduced Units

: mg/L Unit Code : 064000

Supervisor : P. Campbell : Sewage, Industrial Waste, Drinking Waters, Leachates, Effluents and Surface

: Before '81

# SAMPLING:

Quantity Required Container

: 5-500 mL

: Glass or plastic

### ANALYTICAL PROCEDURE:

An appropriate shaken sample volume (5 to 500 mL) is pipetted or quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The graduated cylinder and then the filter are washed with a total of 30 mL distilled water. The filter is dried at 103-105°C, and suspended solids content is calculated by subtracting the original filter mass from the dried filter mass. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

Balance (4/5-decimal places), drying oven, suction filtration apparatus Microcomputer system with appropriate software

## REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

# CALIBRATION:

Balance zero

Internal calibration provided in the balance

#### CONTROLS:

Calibration

:2 S class weights, e.g. OCA for each balance (results in grams)

Recovery

:LTBL plus 2 standards, e.g. R1

Drift Blank :Balance zero is checked every tenth weighing. :Filter washed with 500 mL distilled water:

#### NOTES:

A standard correction factor was applied to all filters to account for weight loss during filtering (-0.0003g). QC data were obtained from two balances. Currently tests on sewage are performed by a private laboratory. QC results reported here represent only tests performed at the Central Laboratory.

# SOLIDS, PARTICULATE

# QUALITY CONTROL DATA FROM 09/01/90 TO 28/12/90

Lab: Solids and BOD

Analytical Range: - to 3000 mg/L

# **CALIBRATION CONTROL:**

	Number of Data	Expected Mass	Av. Mass Measured	Av. Bias	Standard(1) Deviation
		********		********	
A :	124	0.50	0.499989	-0.000011	0.000023
B :	124	0.05	0.049985	-0.000015	0.000026
A+B:	124	0.55	0.549974	-0.000026	0.000040
A-B:	124	0.45	0.450003	0.000003	0.000028

s.d.(AB) S(between runs): 0.000025 Sw(within run): 0.000020 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.54984 - 0.55016 0.44989 - 0.45011

for A+B for A-B

RECOVERIES:

	Number	Expected	Av. Concn	Standard(1)
	of Data	Concn	Measured	Deviation
	*******		*********	***********
R1:	77	200.0	195.4	4.14
R2:	77	50.0	48.5	1.94

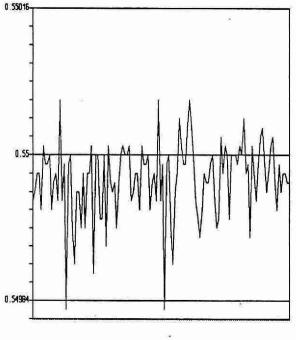
#### **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
	***				
47	0.0	-	5.0	0.5158	17.4
92	5.0		25.0	1.5299	12.2
67	25.0	-940	100.0	3.1773	6.2
30	100.0	-	500.0	17.1702	54.5
11	500.0	1 (get	3000.0	68.5091	6.7
247		Overal	1	2.8269	

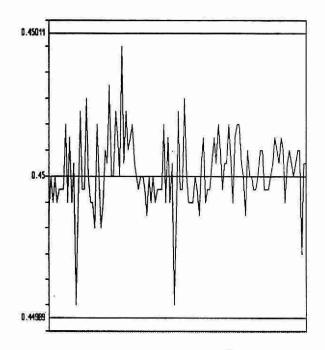
	Number of Data	Data Mean	Standard(1) Deviation
	* * * * * * * * * * * * * * * * * * *	*******	
Blank	138	0.2682	0.7457

# SOLIDS, PARTICULATE (mg/L)

QUALITY CONTROL DATA FROM 09/01/90 TO 28/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

# *** SOLIDS, TOTAL ***

### **IDENTIFICATION:**

Laboratory

: Solids and BOD

Method Introduced

: Before '81

LIS Test Name Code

: RST : SOLIDS Units Unit Code : mg/L or mg/Kg : 064000

Work Station Code Method Code

: 506AB4

Supervisor

: P. Campbell

Sample Type/Matrix

: Sewage, Industrial Waste, Drinking Waters, Leachates, Effluents, Sludge

# SAMPLING:

Quantity Required

: 125 mL

Container

: Glass or plastic

# ANALYTICAL PROCEDURE:

A 50.0 or 100 mL aliquot of sample is pipetted into a preweighed Teflon dish, dried at 103-105°C, and stored in a desiccator for at least 24 hours. After reweighing, the total residue or solids content is calculated by subtracting the original dish mass. Data collection, calculations, and transfer or results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

Balance (4/5 decimal places), drying oven, Teflon dishes Microcomputer system with appropriate software

#### **REPORTING:**

Maximum Significant Figures: 3

Current W value: 2

T value: 10

#### CALIBRATION:

Balance zero

Internal calibration provided in the balance

# CONTROLS:

Calibration

: 2 S class weights, e.g. QCA (results in grams)

Recovery

: BL plus 2 standards, e.g. R1

Drift

: Balance zero is checked every tenth weighing

# NOTES:

Correction factor for dish tare weights, based on variation of a standard sealed vessel, was included in the calculation. QC data were obtained from two balances that are used for all solids analyses. The calibration control data are the same used for Dissolved Solids. Currently tests on sewage are performed by a private laboratory. Insufficient tests were performed at the Central Laboratory to provide a data summary.

#### SULPHATE ***

### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/07/80

LIS Test Name Code

: SSO4FR,SSO4NF

Units Unit Code : ug/Filter as SO4

Work Station Code Method Code

: PRSEQ : 004AI0

Supervisor

: 361941 : F. Lo

Sample Type/Matrix

: Nylon (SSO4NF) filters from LoVol and sequential filter packs, and Teflon

(SSO4FR) filters from sequential filter packs.

# SAMPLING:

Quantity Required

: 1 filter

Container

: 50 mL polypropylene tube

#### SAMPLE PREPARATION:

Filters are extracted with 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03 N NaOH (nylon) in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards. Results are converted to ug/filter as SO₄.

Chloride and sulphate are determined simultaneously.

#### INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 1.0

T value: 5.0

# CALIBRATION:

BL plus 9 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

#### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

# SULPHATE (SSO4FR)

# QUALITY CONTROL DATA FROM 03/01/90 TO 14/12/90

Lab: Ion Chromatography

Analytical Range: - to 250 ug/filter as SO₄

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			*********		
A :	93	200	198.8	-1.2	1.98
B :	93	50	49.8	-0.2	1.18
A+B:	93	250	248.6	-1.4	2.30
A-B:	93	150	149.0	-1.0	2.32

s.d.(AB) S(between runs): 1.63

Sw(within run): 1.64 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

239 - 261 for A+B 143 - 157 for A-B

# **DUPLICATES:**

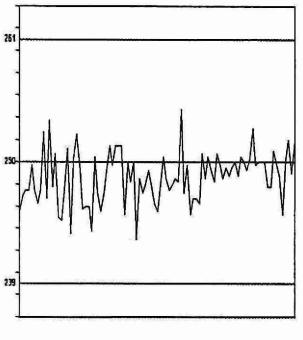
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
				************	
85	0.0	۰ -	25.0	0.559	6.7
123	25.0		100.0	1.014	1.9
43	100.0		250.0	1.669	1.2
251		Overa	11	0.952	

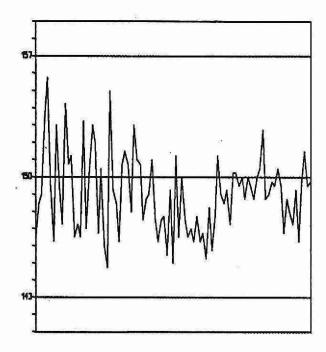
	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	93	0.473	0.935

SULPHATE (ug/filter as SO.)

(SSO4FR)

QUALITY CONTROL DATA FROM 03/01/90 TO 14/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

CONTROL LIMIT

# **SULPHATE** (SSO4NF)

# QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Ion Chromatography

Analytical Range: - to 250 ug/filter as SO₄

# **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
A :	87	200.0	199.5	-0.5	2.16
B :	87	50.0	49.9	-0.1	1.15
A+B:	87	250.0	249.4	-0.6	2.63
A-B:	87	150.0	149.6	-0.4	2.25

s.d.(AB) S(between runs): 1.73 Sw(within run): 1.59 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

261 239 for A+B 143 157 for A-B

# **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
********	****				
88	0		25	0.599	6.1
44	25		50	0.788	2.1
36	50		250	1.027	1.3
168		Overal	I	0.730	

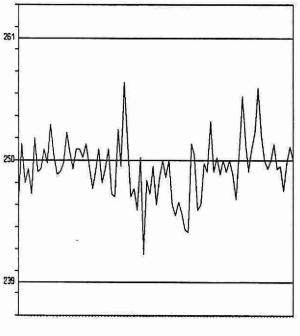
	Number	Data	Standard(1)
	of Data	Mean	Deviation
	***************************************		
Long Term Blank	87	0.132	0.500

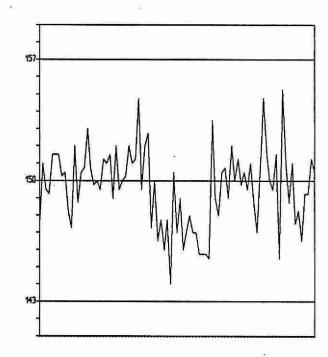
# SULPHATE

(ug/filter as SO,)

(SSO4NF)

QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

_ CONTROL LIMIT

#### *** SULPHATE ***

#### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/78 : mg/L as SO₄

LIS Test Name Code Work Station Code : SSO4UR : PRIC1 : 003AI0 Units
Unit Code
Supervisor

: 064941 : F. Lo

Sample Type/Matrix

: Precipitation, Throughfall, Stemflow

### SAMPLING:

Method Code

Quantity Required

: 15 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards.

Chloride and nitrogen-nitrate are determined simultaneously.

#### INSTRUMENTATION:

Modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing, and partial data processing.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

### NOTES:

Two analytical ranges are in operation at this work station, and subsequently, quality control results are provided for each range.

### **SULPHATE**

### **QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90**

Lab: Ion Chromatography

Analytical Range: - to 5.0 mg/L as SO₄

### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			The state of the s		
<b>A</b> :	45	4.0	4.017	0.017	0.061
B :	45	1.0	0.992	-0.008	0.034
A+B:	45	5.0	5.009	0.009	0.082
<b>A-B</b> :	45	3.0	3.025	0.025	0.056

s.d.(AB) S(between runs): 0.05

Sw(within run): 0.04

S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.77 -2.84 - 5.23 for A+B 3.16 for A-B

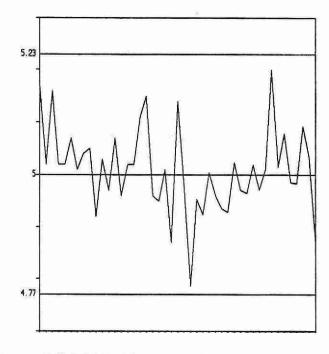
### **DUPLICATES:**

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*****					
. 14	0.0	-	0.5	0.019	37.0
47	0.5	i <del>.</del>	2.0	0.039	3.2
53	2.0		5.0	0.053	2.3
114		Overall		0.043	

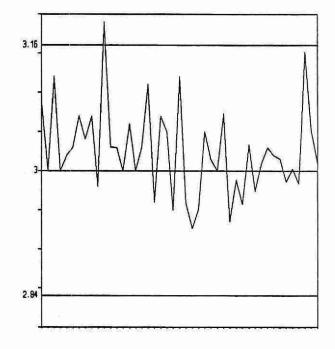
	Number	Data	Standard(1)
	of Data	Mean	Deviation
	********	***********	***************************************
Long Term Blank	45	0.0249	0.040

SULPHATE (mg/L as SO,)

### QUALITY CONTROL DATA FROM 05/01/90 TO 18/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

### **SULPHATE**

### QUALITY CONTROL DATA FROM 11/01/90 TO 28/12/90

Lab: Ion Chromatography

Analytical Range: - to 10.0 mg/L as SO₄

### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			********	* **********	
<b>A</b> :	89	8.0	7.97	-0.03	0.113
B :	89	2.0	1.98	-0.02	0.054
A+B:	89	10.0	9.95	-0.05	0.118
A-B:	89	6.0	5.99	-0.01	0.132

s.d.(AB) S(between runs): 0.089

Sw(within run): 0.093 S/Sw: 0.95

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.4 - 10.6 for A+B 5.6 - 6.4 for A-B

### **DUPLICATES:**

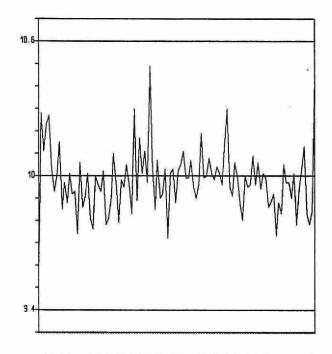
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
					**********
17	0.0		1.0	0.031	12.9
36	1.0	•	5.0	0.056	1.9
153	5.0	-	10.0	0.096	1.6
206	, C	veral!		0.082	COOK SAM

	Number of Data	Data • Mean	Standard(1) Deviation
	,		
Long Term Blank	89	0.0311	0.0601

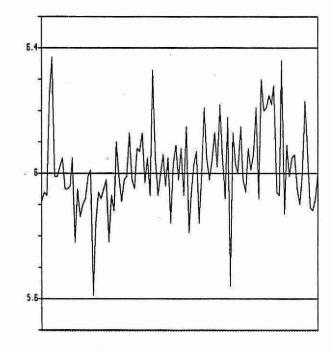
SULPHATE

(mg/L as SO,)

### QUALITY CONTROL DATA FROM 11/01/90 TO 28/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

### *** SULPHATE ***

#### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/78

LIS Test Name Code

: SSO4UR

Units

: ug/Filter as SO.

Work Station Code Method Code

: PRLOV : 004AIC

Unit Code Supervisor : 361941 : F. Lo

Sample Type/Matrix

: W40 filters from LoVol filter packs

### SAMPLING:

**Quantity Required** 

: 1 filter

Container

: 50 mL polypropylene tube

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polypropylene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards. Results are converted to ug/filter as SO₄.

Chloride and nitrogen-nitrate are determined simultaneously.

#### INSTRUMENTATION:

Ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 1.0

T value: 5.0

#### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

#### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

### SULPHATE

### QUALITY CONTROL DATA FROM 19/01/90 TO 20/12/90

Lab: Ion Chromatography

Analytical Range: - to 500 µg/filter as SO₄

### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	********			********	*********
<b>A</b> :	21	400	397.6	-2.4	4.4
B :	21	100	99.6	-0.4	1.5
A+B:	21	500	497.2	-2.8	4.6
<b>A-B</b> :	. 21	300	298.0	-2.0	4.6

s.d.(AB) S(between runs): 3.28 Sw(within run): 3.28

S/Sw: 1.0

On any given day the calibration is accepted if the values obtained lie within the ranges:

478

522 315

for A+B

285

for A-B

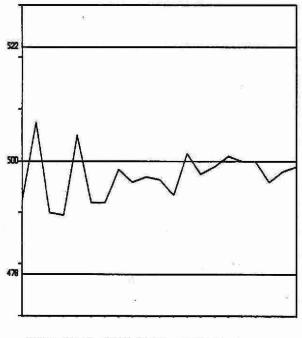
### **DUPLICATES:**

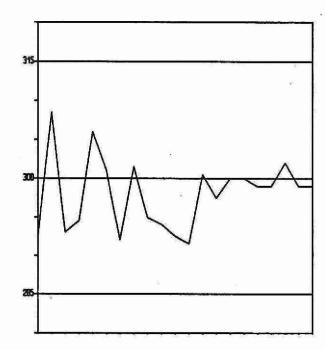
Number of Sample Data Pairs Concn Span			Mean(2) s.d.	Coefficient of var.(%)	
				******	**********
10	0	•	150	1.63	34.8
10	150	-	250	2.03	25.8
5	250	-	500	4.15	13.7
25	Overall			2.24	

	Number of Data	Data Mean	Standard(1) Deviation
	*******		
Long Term Blank	21	0.595	1.751

SULPHATE (ug/filter as SO₄)

### QUALITY CONTROL DATA FROM 19/01/90 TO 20/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

#### *** SULPHATE ***

#### **IDENTIFICATION:**

Laboratory

: Ion Chromatography

Method Introduced

: 01/04/82

LIS Test Name Code

: SSO4UR : RMDSO4 Units

: mg/L as SO₄

Work Station Code Method Code

: 003AI0

Unit Code Supervisor

: 064941 : F. Lo

Sample Type/Matrix

: Rivers, Lakes, Domestic Waters, Leachates, Soil Extracts, Effluents

### SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans.

#### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module (in-house design) for automated sample introduction and timing.

#### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

### **CALIBRATION:**

BL plus 10 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift

: 1 standard every 10 samples

### SULPHATE

### QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90

Lab: Ion Chromatography

Analytical Range: - to 100.0 mg/L as SO4

### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		*********		*********	
<b>A</b> :	158	80.0	80.4	0.4	0.5760
<b>B</b> :	158	20.0	19.9	-0.1	0.4068
A+B:	158	100.0	100.2	0.2	0.8094
<b>A-B</b> :	158	60.0	60.5	0.5	0.5826

s.d.(AB) S(between runs): 0.50

Sw(within run): 0.41 S/Sw: 1.2

On any given day the calibration is accepted if the values obtained lie within the ranges:

96.4 - 103.6 for A+B 57.6 - 62.4 for A-B

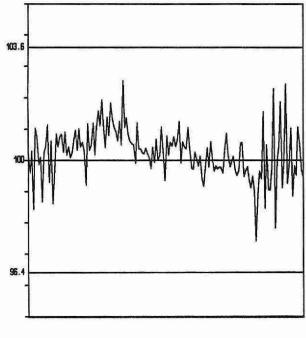
### **DUPLICATES:**

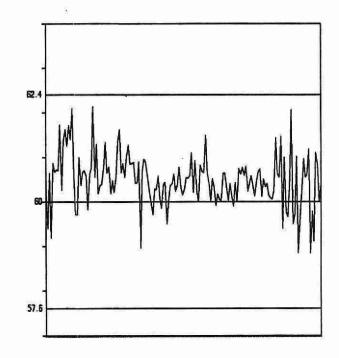
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
*********				*******	***********
168	0.0	4 🗪 2	20.0	0.201	3.1
162	20.0		50.0	0.470	2.1
66	50.0	-	100.0	0.730	1.0
396	(	Overal	1	0.408	

	Number of Data	Data Mean	Standard(1) Deviation
			***************************************
Long Term Blank	158	0.517	0.134

# SULPHATE (mg/L as SO4)

### QUALITY CONTROL DATA FROM 03/01/90 TO 31/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

_____CONTROL LIMIT

### SULPHATE, ORGANO-SULPHUR

#### **IDENTIFICATION:**

Laboratory

LIS Test Name Code

Work Station Code Method Code

Sample Type/Matrix

: Ion Chromatography : SSO4UV

Method Introduced

: 01/03/90 Units : mg/L as SO₄ Unit Code : 064941

Supervisor : Rivers, Precipitation, Soil Extracts

: F. Lo

#### SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

: ORGSO4

: 005IC1

#### ANALYTICAL PROCEDURE:

The aqueous samples are mixed with a dilute solution of hydrogen peroxide and subsequently exposed to ultra-violet light radiation in a continuous flow system. The organo-sulphur compounds are oxidized to sulphate. Sulphate is separated from other anions in the samples by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans.

### **INSTRUMENTATION:**

Basic modular continuous flow ion chromatographic system plus control module (in-house design) for automated sample introduction and timing.

### REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration

: LTBL plus 2 standards, e.g. QCA

Drift : 1 standard every 10 samples

### SULPHATE, ORGANO-SULPHUR

### QUALITY CONTROL DATA FROM 20/03/90 TO 18/12/90

Lab: Ion Chromatography

Analytical Range: - to 20 mg/L as SO4

### **CALIBRATION CONTROL:**

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
<b>A</b> :	46	8.0	8.010	0.010	0.107
B :	46	2.0	2.006	0.006	0.066
A+B:	46	10.0	10.016	0.016	0.148
<b>A-B</b> :	46	6.0	6.004	0.004	0.098

s.d.(AB) S(between runs): 0.09 Sw(within run): 0.07 S/Sw: 1.3

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.45 for A+B 6.30 for A-B 9.55 -5.70 -

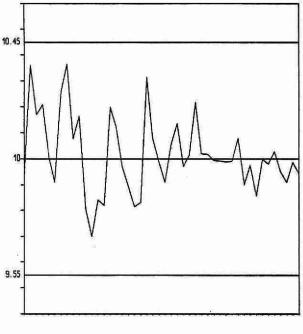
### **DUPLICATES:**

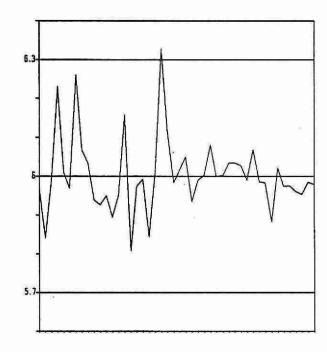
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
				යා කර න න්නි හැකි යා යා යා න	-
9	0	-	1.0	0.012	7.2
20	1.0	-	5.0	0.120	3.9
77	5.0	>=	10.0	0.148	2.1
4	10.0	-	20.0	0.245	2.1
110		Overal	1	0.139	

	Number of Data	Data Mean	Standard(1) Deviation	
	~~~~~			
Long Term Blank	46	< 0.05	0.00	

SULPHATE, ORGANO-SULPHUR (mg/L as SO.)

QUALITY CONTROL DATA FROM 20/03/90 TO 18/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

*** SULPHATE, WATER EXTRACTABLE

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code Method Code Sample Type/Matrix : Dorset Soils

: SSO4EW : DOANIONX : 301AI5

: Soil

Method Introduced

Units Unit Code Supervisor : 01/06/80 : ug/g as SO₄

: 073941 : A. Neary

SAMPLING:

Quantity Required

: 10 g air dried

Container

: Glass

SAMPLE PREPARATION:

Samples are air dried, disaggregated and sieved to <2 mm.

ANALYTICAL PROCEDURE:

Five grams of sample is agitated for 60 minutes with 25 mL deionized water. Samples are centrifuged at 10,000 rpm and the supernatant is filtered through a 0.45 um membrane filter. Sulphate is determined on the filtrate by ion chromatography.

INSTRUMENTATION:

- -Waters Model 430 Conductivity Detector
- -Spectroflow 400 solvent delivery system
- -Spectro-Physics SP780 XR autosampler

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.5

T value: 2.5

CALIBRATION:

BL plus 6 standards; 2 QC solutions at 25% and 75% of full scale.

CONTROLS:

Calibration

: 2 method BL plus 2 standards, e.g. QCA

Recovery Drift

: 2 long term soil samples representing different soil types

: 100% full scale standard every 10 samples

SULPHATE, WATER-EXTRACTABLE

QUALITY CONTROL DATA FROM 05/01/90 TO 26/06/90

Lab: Dorset Soils

Analytical Range: - to 100.0 ug/g

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
		********			***********
A :	14	75.00	73.30	-1.70	2.148
B :	14	36.00	36.35	0.35	1.898
A+B:	14	111.00	109.66	-1.34	3.258
A-B:	14	39.00	36.94	-2.06	2.410

s.d.(AB) S(between runs): 2.02

Sw(within run): 1.70 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

103.5 - 118.5 for A+B 34.0 - 44.0 for A-B

RECOVERIES:

Number of Data		Av. Concn Measured	Standard(1) Deviation	
R1:	17	14.11	0.747	
R2:	17	55.51	1.555	
R3:	17	3.76	2.832	

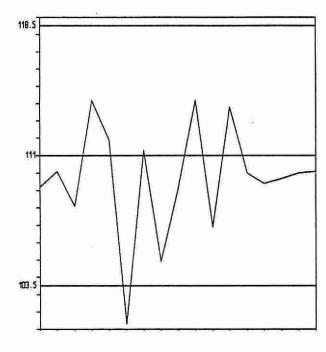
DUPLICATES:

Number of Data Pairs	, ,	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
********				*******	**********
22	0.0	F.	20.0	0.842	7.8
16	20.0	-	50.0	3.473	10.4
11	50.0		100.0	5.212	33.6
49	34 S F00004-38	Overall		2.575	

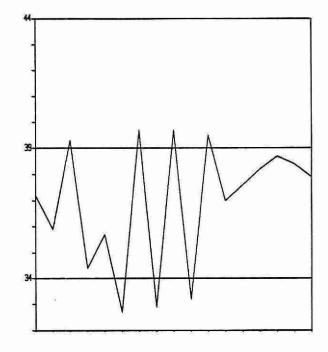
	3	Number of Data	Data Mean	Standard(1) Deviation
			*************	**********
Digested Blank		15	0.393	0.4096

SULPHATE, WATER-EXTRACTABLE (ug/g as SO4)

QUALITY CONTROL DATA FROM 05/01/90 TO 26/06/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

SULPHUR DIOXIDE

IDENTIFICATION:

Laboratory

: Ion Chromatography

Method Introduced

: 01/07/80

LIS Test Name Code

: SSO2FR

Units Unit Code : ug/Filter as SO₂

Work Station Code Method Code

: PRSEQ : 004AI0

: 361943

Supervisor

: F. Lo

Sample Type/Matrix

: W41 filters from LoVol and sequential filter packs.

SAMPLING:

Quantity Required

: 1 filter

Container

: 50 mL polypropylene tube

Other

: Filter is impregnated with potassium carbonate/glycerol solution.

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of 0.05% H₂O₂ in polypropylene tubes with one hour of mechanical shaking, followed by ultrasonic treatment to enhance extraction, then a 24 hour rest period. SO₂ is converted to SO₄ in the process.

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the extract by automated suppressed ion chromatography using an eluent mixture of 0.003 M sodium bicarbonate and 0.0024 M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by the comparison of the sample peak heights to a series of standards. Results are converted to ug/filter as SO₂. Chloride and sulphate are determined simultaneously.

INSTRUMENTATION:

Mechanical shaker, ultrasonic bath; modular continuous flow ion chromatographic system plus microcomputer for automated sample injection, timing and partial data processing.

REPORTING:

Maximum Significant Figures: 3

Current W value: 1.0

T value: 5.0

CALIBRATION:

BL plus 9 standards

CONTROLS:

Calibration

: LTBL plus .2 standards, e.g. OCA

Drift

: 1 standard every 10 samples

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

SULPHUR DIOXIDE

QUALITY CONTROL DATA FROM 03/01/90 TO 20/12/90

Lab: Ion Chromatography

Analytical Range: - to 350 ug/filter as SO2

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	89	268.0	265.7	-2.3	2.73
B:	89	66.0	66.4	0.4	1.35
A+B:	89	334.0	332.1	-1.9	3.21
A-B :	89	202.0	199.3	-2.7	2.88

s.d.(AB) S(between runs): 2.16 Sw(within run): 2.04 S/Sw: 1.1

On any given day the calibration is accepted if the values obtained lie within the ranges:

319 349 192 212 for A-B

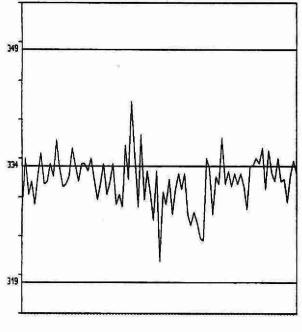
DUPLICATES:

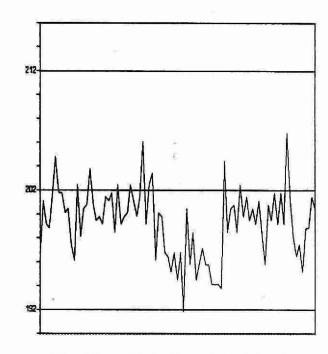
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	ě	Coefficient of var.(%)	
111	0		35	0.498		7.8
23	35	-	70	1.546		2.7
37	70	-	175	1.949		2.2
20	175	-	350	2.169		0.8
191		Overal	1	1.046		

	Number of Data	Data Mean	Standard(1) Deviation
	************	*******	
Long Term Blank	89	0.172	0.659

SULPHUR DIOXIDE (ug/filter as SO₂)

QUALITY CONTROL DATA FROM 03/1/90 TO 20/12/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

*** TURBIDITY ***

IDENTIFICATION:

Laboratory

: Colourimetry

: Ion Chromatography

Method Introduced

: Before '74

LIS Test Name Code

: TURB : RMTURB Units

: FTU

Work Station Code

: KMTURB

Unit Code

: 343000

Method Code

: 002AI1

Supervisor

: P. Campbell

Sample Type/Matrix

: Rivers, Lakes, Effluents

: Drinking Water, Industrial, Sewage

SAMPLING:

Quantity Required

: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

The instrument is standardized with sealed standards which are prepared commercially and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

INSTRUMENTATION:

-Hach Ratio 18900 Turbidimeter

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.05

T value: 0.25

CALIBRATION:

BL plus formazin standards (at least once annually)

CONTROLS:

Calibration

: BL plus two standards, e.g. QCA

NOTES:

Two workstations are used for turbidity analyses: RMTURB and WTURB. Each used a different set of standards for calibration control. Laboratory, Work Station Code, Sample Type/Matrix, are given in their respective orders.

TURBIDITY

QUALITY CONTROL DATA FROM 02/01/90 TO 22/08/90

Lab: Colourimetry

Analytical Range: - to 200 FTU

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
			**********	*********	
A :	130	18.0	17.90	-0.10	0.0826
B :	130	1.8	1.74	-0.06	0.0453
A+B:	130	19.8	19.64	-0.16	0.0950
A-B :	130	16.2	16.16	-0.04	0.0933

s.d.(AB) S(between runs): 0.067

Sw(within run): 0.066 S/Sw: 1.0

On any given day the calibration is accepted if the values obtained lie within the ranges:

18.6 - 21.0 for A+B 15.4 - 17.0 for A-B

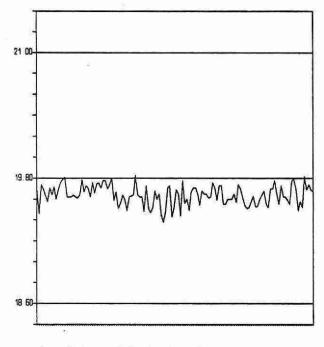
DUPLICATES:

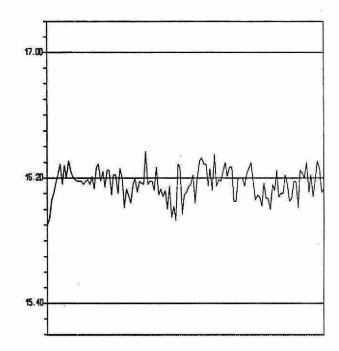
Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	
				********	***************************************
29	0.0		1.0	0.0648	24.3
49	1.0	-	2.0	0.1264	10.4
115	2.0	n-	10.0	0.2645	7.7
51	10.0	194	50.0	0.7758	5.7
18	50.0	15	200.0	2.1423	2.4
262		Overa	1	0.3352	

	Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	130	0.098	0.231

TURBIDITY (FTU)

QUALITY CONTROL DATA FROM 02/01/90 TO 22/08/90





QUALITY CONTROL STANDARD A+B

QUALITY CONTROL STANDARD A-B

_____ CONTROL LIMIT

TURBIDITY

QUALITY CONTROL DATA FROM 03/01/90 TO 21/12/90

Lab: Titration

Analytical Range: - to 200 FTU

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation

A :	209	18.00	18.1	0.10	0.2135
B :	209	1.80	1.75	-0.05	0.0295
A+B:	209	19.80	19.85	0.05	0.2349
A-B:	209	16.20	16.35	0.15	0.1942

s.d.(AB) S(between runs): 0.15

Sw(within run): 0.14 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

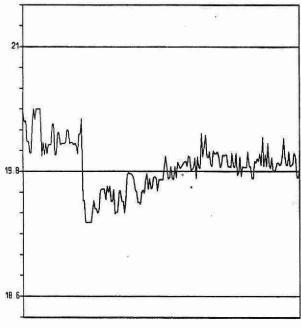
18.6 - 21.0 for A+B 15.4 - 17.0 for A-B

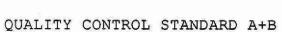
DUPLICATES:

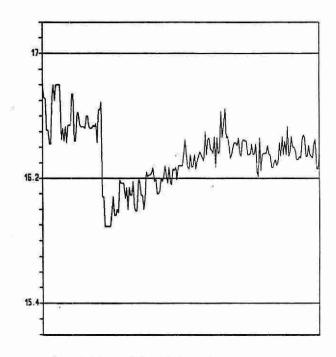
Number of Data Pairs	THE PERSON NAMED OF THE PE		Mean(2) s.d.	Coefficient of var.(%)	
215	0.0		1.0	0.0293	7.2
54	1.0	74	2.0	0.0608	5.1
79	2.0	**	10.0	0.1740	6.0
34	10.0	-	50.0	0.9024	4.1
12	50.0		200.0	2.3978	2.8
394	(Overa	11	0.0816	

TURBIDITY (FTU)

QUALITY CONTROL DATA FROM 03/01/90 TO 21/12/90







QUALITY CONTROL STANDARD A-B

*** ZINC, ACID EXTRACTABLE ***

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code Method Code

Sample Type/Matrix

: Dorset Soils Method Introduced

Units Unit Code

: ug/g as Zn : 073830 Supervisor : A. Neary

: 01/06/80

SAMPLING:

Container

Quantity Required

: 1 g dry : Glass

: ZNUT

: Soil

: DOHMTE

: 551AA1

SAMPLE PREPARATION:

Samples are air dried, disaggregated, and sieved to < 2 mm. A subsample is ground to < 500 um (35 mesh).

ANALYTICAL PROCEDURE:

A 0.500 g sample plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125°C for 2 hours. The temperature is increased to 175°C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, mixed using a Vortex mixer and allowed to settle and decanted. The supernatant is analyzed for Zn by AAS at 217.0 nm using an air - acetylene flame.

Approximate absorbance: 0.3 at the full scale value.

Copper, nickel and zinc are also determined on the extract.

INSTRUMENTATION:

-Varian AA1275 with programmable sample changer and Gilson Minipuls II pump

-Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3

Current W value: 0.5

T value: 2.5

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration

:Three long term soil samples representing different soil types, 2 method blanks, and one

judiciously blended sample digest run with each run.

Drift

:BL plus 1 standard (100% F.S.) every 10 samples

NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal. Values for recoveries are unknown - average value used.

ZINC, ACID EXTRACTABLE

QUALITY CONTROL DATA FROM 19/06/90 TO 21/11/90

Lab: Dorset Soils

Analytical Range: - to 100.0 ug/g as Zn

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard(1) Deviation
	*********	********			
A :	4	79.0	80.20	0.20	2.667
B :	4	30.0	31.05	1.05	1.894
A+B:	4	109.0	111.25	2.25	3.923
A-B:	4	49.0	49.15	0.15	1.453

s.d.(AB) S(between runs): 2.31

Sw(within run): 1.73 S/Sw: 1.33

On any given day the calibration is accepted if the values obtained lie within the ranges:

101.5 - 116.5 for A+B 44.0 - 54.0 for A-B

RECOVERIES:

*	Number of Data	Av. Concn Measured	Standard(1) Deviation

R1:	4	35.2	1.716
R2:	4	79.6	1.831
R3:	4	39.82	2.007

DUPLICATES:

Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)	

4	0.0		20.0	0.933	5.9
6	20.0	-	50.0	1.247	3.6
2	50.0	•	100.0	N.A	N.A
12		Overa	11	1.103	

	Number of Data	Data Mean	Standard(1) Deviation

Digested Blank	4	1,25	0.900

*** ZINC, TOTAL ***

IDENTIFICATION:

Laboratory

LIS Test Name Code

Work Station Code Method Code Sample Type/Matrix : Dorset

: ZNUT : DOASV

: 001PP2 : Streams, Lakes, Precipitation Method Introduced Units

: 01/03/86 : ug/L as Zn : 063830

Unit Code Supervisor

: A. Neary

SAMPLING:

Quantity Required

: 100 mL

Container

: 500 mL, acid washed Teflon container, bagged in a clean room

ANALYTICAL PROCEDURE:

Samples are acidified to 0.1% using Seastar nitric acid in a clean room. Oxygen is removed by nitrogen gas and samples are analyzed using anodic stripping voltammetry on a hanging mercury drop electrode. Change in current when zinc is stripped from mercury drop is proportional to concentration.

INSTRUMENTATION:

Metrohm 646 VA Processor with Model 675 VA Sample Changer.

REPORTING:

Maximum Significant Figures: 3

Calculated W value: 0.5

T value: 2.5

CALIBRATION:

BL plus 2 standards daily

CONTROLS:

Calibration Duplicate

: LTBL plus 2 standards, e.g. QCA + EPA standard. : End of every run (approximately every 8 samples)

ZINC, TOTAL

QUALITY CONTROL DATA FROM 04/01/90 TO 18/12/90

Lab: Dorset

Analytical Range: - to 15.00 ug/L as Zn

CALIBRATION CONTROL:

	Number	Expected	Av. Conen	Av.	Standard(1)
	of Data	Concn	Measured	Bias	Deviation
A :	45	8.00	9.58	1.58	1.21
B :	45	2.00	2.31	0.31	0.52
A+B:	45	10.00	11.90	1.90	1.27
A-B:	45	6.00	7.27	1.27	1.36

s.d.(AB) S(between runs): 0.93

Sw(within run): 0.96 S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within the ranges:

5.50 - 14.50 for A+B 3.00 - 9.00 for A-B

DUPLICATES:

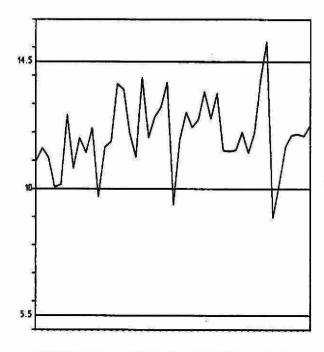
Number of Data Pairs	C	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)

9	0.0	-0	5.0	0.436	17.4
6	5.0	-	10.0	0.650	11.0
4	10.0	-0	15.0	2.216	16.3
19	(Overal	1	0.891	

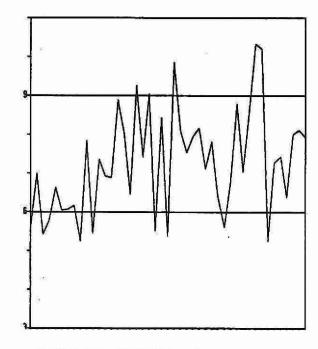
	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	45	0.037	0.124

ZINC, TOTAL (ug/L as 2n)

QUALITY CONTROL DATA FROM 04/01/90 TO 18/12/90



QUALITY CONTROL STANDARD A+B



QUALITY CONTROL STANDARD A-B

PART 3.0

MICROBIOLOGY

3.1 Quality Control Program, Microbiology

Sources of error in a microbiological lab often are attributed to lack of implementation of quality control procedures for media preparation and storage, equipment malfunction, inadequate cleaning or sterilization of glassware and impure water supplies. Detailed information regarding the quality control program for these issues are discussed in the LSB publications (8)(9). This report discusses only the quality control procedures that are related directly to sample analysis.

Analyses on microbiological samples for bacteria indicative of pollution require the careful use of methods and techniques by technicians to prevent contamination which will produce either false positive or false negative results. Checks are made to ensure that the analytical procedures are functioning properly and providing the client with results that are both accurate and reproducible within the limits of normal statistical variation. To this end, a series of quality control tests are conducted on a regular basis and their results are monitored so that any irregularities in the test procedures are corrected and false results are not reported.

Membrane Filtration Test (MF)

Blank Control Filters

Each sample analyzed by the membrane filter test is separated from the previous sample by introducing a control filter at the beginning of each analysis. The control filter is employed in the same manner as those filters used for sample analyses, however, only sterile buffered rinse water is filtered. The control filter is placed on the same bacterial medium as used for incubation of filters from the actual sample, so that all filters are incubated under the same conditions. If any bacteria appear on the control filter, they were likely carried over from the previous sample. No target or indicator organisms and <10 non-target or background organisms should appear on the control filter. If these limits are exceeded, the senior technician/supervisor is consulted. If excessive contamination is suspected the result will not be reported and, if possible, the analyses will be repeated.

Duplicate Analyses

Duplicate analyses are conducted at a frequency of one in twenty samples. The data are accumulated for each parameter and a within-run standard deviation is calculated to give a measure of the repeatability of results. The calculation of the standard deviation is the same as that used in section 2.1 under sample repeatability. A control limit is established based on historical data whereby, the observed differences in duplicate results are sorted according to ranges of colony counts per filter. At present three ranges are used: 0 - 30, 31 - 75, and 76 - 150 colonies per filter. The mean difference within each range is multiplied by 3.267 (3). If the control limit is exceeded the senior technician/supervisor is consulted. If excessive bias is suspected, the results will not be reported.

Media Quality Control

A number of checks are made both during and after the preparation of a batch of medium. The pH of a medium is monitored after all the ingredients have been added and again after sterilization has taken place. The final pH may vary within ±0.2 units from the recommended value. The medium is checked for sterility at both 20°C and 35°C by incubating random samples of either tubes or plates depending on how the medium is dispensed. Any bacterial growth will require retesting of the medium for sterility. Confirmation of contamination will result in the rejection of the medium for any further use. The batch or lot number of a medium is recorded to determine if any changes in quality occur when batch or lot numbers change.

Differential agar media used in the detection and enumeration of indicator bacteria are tested to ensure their proper functioning. The medium is streaked with both a known target organism or positive culture and a known non-target organism or negative culture. If the medium is functioning properly, growth of the target organism will be abundant after 24 to 48 hours and growth of the non-target organism will not occur or will be minimal even after 72 hours of incubation. The results of all such tests are recorded and any deviations from the expected results will require retesting or rejection of the medium.

A quantitative QC test of agar media for membrane filter tests involves making up dilute suspensions of the positive and negative cultures. Selected dilutions of these suspensions are passed through membrane filters, which are then placed on plates of both the inhibitory or selective medium and plates of a noninhibitory or nonselective medium, such as Brain Heart Infusion agar. The positive culture should form approximately the same number of colonies on the selective and nonselective media plates, whereas the negative culture should only form colonies on the nonselective medium. This procedure is conducted on one out of ten media batches. Alternatively, one or more samples containing target organisms are filtered in duplicate and respective filters are placed on agar plates from the new and previous batch of medium. Results are recorded and statistically analyzed as for duplicate analyses. Media is retested and rejected if it fails to meet the past performance of the previous medium. Results are recorded and statistically analyzed in a manner similar to that for duplicate analyses.

Presence-Absence (P-A) Tests

Blank Control P-A Bottles

For each group of 21 samples, a blank control is prepared by pouring a 99 mL dilution blank into a P-A bottle and incubating it along with the regular P-A bottles. The P-A blank bottle is incubated for three to four days and should remain free of any bacterial growth or colour change. Growth in more than one P-A blank control test will require rechecking of the sterility of the dilution blanks and P-A medium.

Media Ouality Control

A number of checks are performed on the P-A broth including: pH, sterility at 20°C and 35°C, and growth reaction of <u>Escherichia coli</u>. If the medium is functioning properly, <u>E. coli</u> will produce a strong acid reaction (yellow colour in the medium) and agitation of the bottle will cause release of the dissolved gas in the medium producing a layer of foam at its surface.

In addition, a quantitative test of the P-A broth is done by pipetting 2 mL of broth onto a filter pad and filtering a suspension of <u>E. coli</u> through a membrane filter, which is then placed on the filter pad saturated with the P-A broth. A second MF is prepared with a similar volume of an <u>E. coli</u> suspension and placed on Brain Heart Infusion agar in a petri dish. Previous testing has shown that <u>E. coli</u> colony counts on both filters should be approximately the same. Quality Control checks on EC broth, Lactose Purple broth, MacConkey agar, Nutrient Gelatin Yeast Extract agar and Mannitol Salt agar include pH readings, sterility and bacterial growth reactions of positive and negative cultures inoculated onto or into each medium. If any Quality Control tests fail, the medium is either retested or rejected.

Sample Age

The accurate determination of bacterial numbers for indicator or heterotrophic bacteria in a sample depends on how quickly the sample can be transported to the laboratory for analysis. Water samples should be kept as close to the original water temperature by using a foam-pack container, which includes a central plastic bottle containing water that has been frozen, or by cooling to refrigeration temperatures before shipment to the laboratory. (10)

Samples should arrive at the laboratory on the same day as sampled or, if refrigerated, within 24 hours. For sewage effluent and surface water samples, no analysis will be done if the samples are older than 48 hours; for drinking water samples, the time limit is 72 hours, and for legal samples, it is 24 hours. Limits on the age of samples for analysis must be in place as bacterial numbers in samples may increase or decrease depending on nutrients, toxic elements and the influence of temperature on the metabolic activities of the organisms. The longer the time period between sampling and analysis, the greater the chance for producing either inflated or deflated numbers of organisms per 100 mL of sample.

3.2 PERFORMANCE SUMMARIES MICROBIOLOGY

ESCHERICHIA COLI

IDENTIFICATION:

Laboratory : Surface and Waste Method Introduced

: 1979 Waters

LIS Test Name Code : ECMF Units : Counts/100mL

Work Station Code : MSBACIND Unit Code : 301532 Method Code : TFC 24 Supervisor : J. Clark

Sample Type/Matrix : Surface and Waste Waters

SAMPLING:

Quantity Required : 100 mL

Container : 250 mL glass or plastic Preservative : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mTEC agar and incubated for 23 +/- 1 hour at 44.5 +/- 0.5°C to allow for colony development. The temperature is gradually elevated by incubating 10 plates (2 stacks of 5 plates placed in the centre of a plastic container with lid) with 2 plastic jars containing ice (50 mL of water), one placed at each end of the plastic container. After incubation the membrane filter is transferred to a pad soaked in urease reagent and is given a reaction time of 15 minutes. All colonies that were yellow on mTEC agar and remain yellow on urease are counted as E. coli. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium OC : Target organism count on selective medium vs nonselective medium.

: Comparison of target counts on an old vs a new batch.

Escherichia coli

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Surface and Waste Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number of Data			Mean difference	Standard dev (2)	Coefficient of var. (%)
	NO AC do do de			*******	***********
55	0 -	30	3.2	2.9	19.3
19	31 -	75	6.2	5.9	11.1
7	76 -	150	10.0	8.7	7.7

CONTROL FILTERS:

Number	Number of	Percent
of controls	positive controls	positive

1734	0	0.0

MEDIUM QC: mTEC agar (previous batch) vs mTEC agar (new batch) - inoculated with surface or waste water sample.

Number of Data	Counts per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)
18	0	- 30	3.2	3.0	20.0
40	31	- 75	7.6	6.4	11.4
6	76	- 150	8.3	7.2	6.4

ESCHERICHIA COLI

IDENTIFICATION:

: Surface and Waste Laboratory

Method Introduced

: May 1, 1986

LIS Test Name Code

Waters : ECMMF

Units

: Counts/100mL

Work Station Code

: MSBACIND

Unit Code

: 301532

Method Code Sample Type/Matrix : TGM 24

Supervisor : Waste Waters eg. Pulp and Paper effluent.

: J. Clark

SAMPLING:

Quantity Required

: 100 mL

Container Preservative : 250 mL glass or plastic : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mTEC MUG agar and incubated for 21 +/- 1 hours at 44.5 +/- 0.5°C to allow for colony development. The temperature is gradually elevated by incubating 10 plates (2 stacks of 5 plates placed in the centre of a plastic container with lid) with 2 plastic jars containing ice (50 mL of water), one placed at each end of the plastic container. Under UV light (long wave 366nm) all blue fluorescent colonies are counted as E. coli. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium OC

: Target organism count on selective vs nonselective medium.

*** FECAL COLIFORMS ***

IDENTIFICATION:

Laboratory : Surface and Waste Method Introduced

: April 1979

Water

: Municipal Drinking

Water

LIS Test Name Code : FCMF

Units

: Counts/100mL

Work Station Code

: MSBACIND : WQMFPA

Unit Code

: 301532

Method Code

: TF1 24

Supervisor

: J. Clark

Sample Type/Matrix

: Surface, Waste and Drinking Waters

SAMPLING:

Quantity Required

: 100 mL

Container Preservative : 250 mL glass or plastic : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mTEC agar and incubated for 23 +/- 1 hours at 44.5 +/- 0.5°C to allow for colony development. The temperature is gradually elevated by incubating 10 plates (2 stacks of 5 plates placed in the centre of a plastic container with lid) with 2 plastic jars containing ice (50 mL of water), one placed at each end of the plastic container. All yellow, yellow brown, and yellow green colonies are counted as fecal coliforms. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium OC : Target organism count on selective medium vs nonselective medium.

FECAL COLIFORMS

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Surface and Waste Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number of Data	Counts per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)	
	******			***********		
169	0 -	30	2.7	2.7	18.0	
68	31 -	75	6.1	5.6	10.6	
53	76 -	150	8.8	7.7	6.8	

CONTROL FILTERS:

Number of controls	Number of positive controls	Percent positive	
	F	positive	
4803	21	0.44	

MEDIUM QC: mTEC agar (previous batch) vs m TEC agar (new batch) - inoculated with surface or waste water sample.

Number of Data	Counts per filter			Mean difference	Standard dev (2)	Coefficient of var. (%)	

18	0	A 1	30	3.2	3.0	20.0	
40	31	-	75	7.6	6.4	11.4	
6	76	*	150	8.3	7.2	6.4	

FECAL COLIFORMS

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Municipal Drinking Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number of Data		ints filter	Mean difference	Standard dev (2)	Coefficient of var. (%)	

99	0 -	30	2.4	2.3	15.3	
17	31 -	75	8.8	7.3	11.9	
5	76 -	150	7.8	5.8	5.1	

CONTROL FILTERS:

Number	Number of	Percent
of controls	positive controls	positive
		4 (************************************
N.A	N.A	N.A

MEDIUM QC: mTEC agar LES (Selective) vs Brain Heart Infusion agar (Non-selective) Test organism - <u>E. coli</u>

Number of Data	Counts per filter			Mean difference	Standard dev (2)	Coefficient of var. (%)	
18	0		30	3.2	3.0	20.0	
40	31	-	75	7.6	6.4	11.4	
6	76	-	150	8.3	7.2	6.4	
						N.A	

*** FECAL COLIFORMS ***

IDENTIFICATION:

Laboratory: Surface and Waste Method Introduced: May 1, 1986

Waters

LIS Test Name Code : FCMMF Units : Counts/100mL

Work Station Code : MSBACIND Unit Code : 301532 Method Code : TGM 24 Supervisor : J. Clark

Sample Type/Matrix : Waste Waters eg. Pulp and Paper effluent.

SAMPLING:

Quantity Required : 100 mL

Container : 250 mL glass or plastic Preservative : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mTEC MUG agar and incubated for 21 +/- 1 hours at 44.5 +/- 0.5°C to allow for colony development. The temperature is gradually elevated by incubating 10 plates (2 stacks of 5 plates placed in the centre of a plastic container with lid) with 2 plastic jars containing ice (50 mL of water), one placed at each end of the plastic container. After the <u>E. coli</u> count is determined the filters are removed from mTEC MUG agar and placed onto mTEC agar. The plates are reincubated at 44.5° C for 1 hour (no ice is required). All yellow, yellow brown, and yellow green colonies are counted as fecal coliforms. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium QC : Target organism count on selective vs nonselective medium.

*** FECAL STREPTOCOCCUS ***

IDENTIFICATION:

Laboratory

: Surface and Waste

Method Introduced

: Apr. 1972

LIS Test Name Code

Waters : FSMF

Units

: Counts/100mL

Work Station Code Method Code

: MSBACIND

Unit Code

: 301532

: EF 48

Supervisor

: J. Clark

Sample Type/Matrix

: Surface and Waste Waters

SAMPLING:

Quantity Required

: 100 mL

Container Preservative : 250 ml glass or plastic : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mEnterococcus agar and incubated for 48 +/- 3 hours at 35 +/- 0.5°C to allow for colony development. All colonies that are red, maroon or pink are counted as fecal streptococcus. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium QC

: Target organism count on selective medium vs non selective medium

FECAL STREPTOCOCCUS

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Surface and Waste Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number of Data	Counts per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)	
	====					
148	0 -	30	2.7	2.5	16.7	
72	31 -	75	6.5	6.0	11.3	
27	76 -	150	8.0	6.4	5.7	

CONTROL FILTERS:

Number	Number of	Percent
of controls	positive controls	positive
N.A	N.A	N.A

MEDIUM QC: mEnterococcus agar (previous batch) vs m Enterococcus agar (new batch) - inoculated with surface or waste water sample.

Number of Data	p	Counts per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)	
			***		********		
8	0	-	30	2.9	2.5	16.6	
19	31		75	4.5	4.3	7.6	
5	76		150	9.8	8.1	7.1	

*** HETEROTROPHS ***

IDENTIFICATION:

Laboratory

: Surface and Waste

Method Introduced

: April 1, 1979

Water

: Municipal Drinking

Water

LIS Test Name Code Work Station Code

: **HB35MF** : MSBACIND Units

: Counts/mL

: WOMFPA

Unit Code

: 301532

Method Code

: SF 48

Supervisor

: J. Clark

Sample Type/Matrix

: Drinking Water

SAMPLING:

Quantity Required

: 100 mL

Container Preservative : 250 mL glass or plastic : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mHPC agar and incubated for 48 +/- 3 hours at 35 +/- 0.5°C to allow for colony development. All colonies are counted as heterotrophs. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium QC

: Colony counts are obtained using a pure culture and comparing the heterotrophic medium

(mHPC) to the nonselective medium (BHIA).

: Comparison of colony counts on an old vs a new batch are done using water samples.

HETEROTROPHS

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Surface and Waste Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number of Data		ounts filter	Mean difference	Standard dev (2)	Coefficient of var. (%)	
62	0 -	30	2.7	2.8	18.7	
3	31 -	75	11.3	11.0	20.8	
4	76 -	150	5.5	4.9	4.3	

CONTROL FILTERS:

Number	Number of	Percent
of controls	positive controls	positive
1229	147	12.0

MEDIUM QC: mSPCI agar (previous batch) vs mSPCI agar (new batch) - inoculated with surface or drinking water sample.

Number of Data		Counts per filter		Mean difference		Standard dev (2)		Coefficient of var. (%)	
						-			
10	0	- 1	30	2.0			1.7	11.4	
4	31		75	7.8			5.8	10.4	
N.A.	76	- 13	50	N.A			N.A	N.A	

HETEROTROPHS

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Municipal Drinking Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number Cou of Data per fi			Mean difference	Standard dev (2)	Coefficient of var. (%)	

115	0	•	30	2.4	2.3	15.3
18	31	-	75	7.8	6.7	12.6
8	76	•	150	6.6	5.0	4.4

CONTROL FILTERS:

Number	Number of	Percent
of controls	positive controls	positive

1339	170	12.7

MEDIUM QC: mSPCI agar (previous batch) vs mSPCI agar (new batch) - inoculated with surface or drinking water sample.

Number of Data	Counts per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)

10	0	- 30	2.0	1.7	11.4
4	31	- 75	7.8	5.8	10.4
N.A.	76	- 150	N.A	N.A	N.A

*** PRESENCE-ABSENCE (P-A) TEST ***

IDENTIFICATION:

Laboratory

: Municipal Drinking

Method Introduced

: 1968

LIS Test Name Code

: PABOT

Units:Present/Absent/100mL Unit Code

: 999000

Work Station Code Method Code

: WQMFPA : LLSB10

Water

Supervisor

: J. Clark

Sample Type/Matrix

: Drinking Water

SAMPLING:

Ouantity Required

: 100 mL

Container Preservative

: 250 mL glass or plastic : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

A 100 mL volume of sample is added to a presence-absence (P-A) bottle. The bottle is incubated at 35°C for 3 to 4 days and examined every 24 hours for acid or acid and gas formation. When a positive reaction for acid or acid and gas occurs, the inoculum is transferred to confirmatory media to determine the presence of total coliforms, fecal coliforms and other indicator organisms.

REPORTING:

Microbiological parameters are reported either as present or absent per 100 mL of sample.

CONTROLS:

: A blank control sample is included for every 20 to 25 samples.

Medium QC

: P-A broth batches are checked for sterility at 20° and 35°C and inoculation of the medium is done with E. coli to determine its response. Dilutions of E. coli are passed through membrane filters which are subsequently placed on filter pads saturated with P-A broth and on an enrichment medium, such as Brain Heart Infusion Agar, to compare numbers of colonies recovered.

PRESENCE-ABSENCE (P-A) TEST

QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Municipal Drinking Water

CONTROL FILTERS:

Number	Number of	Number of	Percent
of samples	controls	positive controls	positive controls
		·	
22353	1142	6	0.53

MEDIUM QC: P-A Broth (Selective) vs Brain Heart Infusion agar (Non - selective) Test Organism - <u>E. coli</u>

Number of Data		unts filter	Mean difference	Standard dev (2)	Coefficient of var. (%)
	**********		****		
66	0 -	30	3.0	3.0	19.7
33	31 -	75	6.5	6.5	11.6
26	76 -	150	15.6	14.0	12.4

PSEUDOMONAS AERUGINOSA

IDENTIFICATION:

Laboratory : Surface and Waste Method Introduced : May 1980

Waters

LIS Test Name Code : PSAMF Units : Counts/100mL

Work Station Code : MSBACIND Unit Code : 301532 Method Code : PF 48 Supervisor : J. Clark

Sample Type/Matrix : Surface and Waste Waters

SAMPLING:

Quantity Required : 100 mL

Container : 250 mL glass or plastic Preservative : Sodium Thiosulphate

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mPA agar and incubated for 48 +/- 2 hours at 41.5 +/- 0.5°C to allow for colony development. All colonies that are dark brown, brown with darkened centers, tan and usually very flat in appearance are counted as <u>Pseudomonas aeruginosa</u>. An ideal counting range is 10 to 100 colonies per filter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

CONTROLS:

Duplicate samples and blank filter between each sample.

Medium OC : Target organism count on selective medium vs nonselective

meaium.

PSEUDOMONAS AERUGINOSA

QUALITY CONTROL DATA FROM 01/01/90 TO 01/12/90

Lab: Surface and Waste Water

Analytical Range: 0 to 150 counts/filter

DUPLICATES: Within-run precision

Number of Data			Mean difference	Standard dev (2)	Coefficient of var. (%)	
63	0		30	2.4	2.2	14.7
14	31	-	75	5.1	4.5	8.5
5	76	-	150	7.2	6.0	5.3

CONTROL FILTERS:

Number	Number of	Percent
of controls	positive controls	positive
~~~~~~~~~~~~~~~~	***************************************	
N.A	N.A	N.A

MEDIUM QC: mPA agar (previous batch) vs mPA agar (new batch) - inoculated with surface or waste water sample.

Number of Data		Counts per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)
			***			
0	0	-	30	N.A	N.A	N.A
0	31	l <del>e</del>	75	N.A	N.A	N.A
0	76	-	150	N.A	N.A	N.A

### *** TOTAL COLIFORMS ***

### **IDENTIFICATION:**

Laboratory : Surface and Waste

Method Introduced

: Jan. 1971

Water

: Municipal Drinking

Water

LIS Test Name Code

: TCMF

Units

: Counts/100mL

Work Station Code

: MSBACIND

Unit Code

: 301532

Work Station Code

: WQMFPA : LF 22

Supervisor

: J. Clark

Sample Type/Matrix

: Surface Water, Drinking Water

#### SAMPLING:

Method Code

Quantity Required

: 100 mL

Container Preservative 250 mL glass or plasticSodium Thiosulphate

### ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mENDO LES agar and incubated for 22 +/- 2 hours at 35 +/- 0.5°C to allow for colony development. All colonies with a dull to bright metallic green-gold sheen are counted as coliforms. An ideal counting range is 10 to 100 colonies per filter.

### REPORTING:

Maximum Significant Figures: 3

Minimum Increment: 1 Detection Criteria: 10

### CONTROLS:

: Duplicate samples and blank filter between each sample.

Medium QC

: Target organism count on selective medium vs nonselective medium.

# TOTAL COLIFORMS

# QUALITY CONTROL DATA FROM 13/02/89 TO 28/12/89

Lab: Surface and Waste Water

Analytical Range: 0 to 150 counts/filter

**DUPLICATES:** Within-run precision

Number of Data			Mean difference	Standard dev (2)	Coefficient of var. (%)	
58	0	-	30	2.2	2.2	14.7
11	31	-	75	8.2	6.9	13.0
7	76	-	150	4.9	4.7	4.1

# **CONTROL FILTERS:**

Number	Number of	Percent
of controls	positive controls	positive
	****	
643	14	2.2

MEDIUM QC: mEndo agar LES (Selective) vs Brain Heart Infusion agar (Non-selective) Test organism - <u>E. coli</u>

Number of Data	of Data per filter			Mean difference	Standard dev (2)	Coefficient of var. (%)
					*****	
33	0	-	30	3.1	2.7	18.2
14	31		75	5.9	4.9	8.7
12	76	-	150	10.5	8.2	7.2

# TOTAL COLIFORMS

# QUALITY CONTROL DATA FROM 01/01/90 TO 31/12/90

Lab: Municipal Drinking Water

Analytical Range: 0 to 150 counts/filter

**DUPLICATES:** Within-run precision

Number of Data	Counts per filter	Mean difference	Standard dev (2)	Coefficient of var. (%)
	*******			
112	0 - 30	2.8	2.6	17.5
24	31 - 75	7.1	6.4	12.1
11	76 - 150	11.7	10.5	9.3

# **CONTROL FILTERS:**

Number	Number of	Percent
of controls	positive controls	positive
4036	108	2.7

MEDIUM QC: mEndo agar LES (Selective) vs Brain Heart Infusion agar (Non-selective) Test organism - <u>E. coli</u>

Number Counts of Data per filter		Mean difference	Standard dev (2)	Coefficient of var. (%)	
				********	
33	0 -	30	3.1	2.7	18.2
14	31 -	75	5.9	4.9	8.7
12	76 -	150	10.5	8.2	7.2

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### **ABBREVIATIONS**

AAS

- Atomic Absorption Spectrophotometer

Abs

- Absorbance

**APIOS** 

- Acidic Precipitation in Ontario Study

Av

- Average

Bl

- Blank

C

- Degrees Centigrade

cm

- Centimeter

Concn

- Concentration

Date

- Day/Month/Year

DDW

- Deionized, distilled water

DO

- Dissolved oxygen

DW

- Distilled water

**ECSS** 

- Expert Committee on Soil Survey (Land Resource Research

Centre)

**EPA** 

- Environmental Protection Agency

FTU

- Formazin Turbidity Units

g

- Gram

HOAC

- Acetic Acid

HZU

- Hazen Units

IR

- Infra-Red

kg

Kilogram

L

- Litre

LAB

- Laboratory

LIS

- Laboratory Information System

LTBL

Long Term Blank

M

- Molar

meq

- Milliequivalent

mg

Milligram

min

- Minute

### ABBREVIATIONS cont'd

mL - Millilitre mm - Millimeter

N.A. - Not Available or Not Applicable

nm - Nanometer

QC - Quality Control

QCA - Quality Control Standard A
QCB - Quality Control Standard B
QCC - Quality Control Standard C

QCD - Quality Control Standard D

R - Recovery

rpm - Revolutions per minute

S - Between run standard deviation for QC

S₁ - Standard deviation

S₂ - Standard deviation for duplicates

S_w - Within run standard deviation for QC

S. Class - Weights that have not been certified .

s.d. - Standard deviation

Standard Cal - Colourimeter setting to control electronic expansion

STD - Standard

TCU - True Colour Units

um - Micrometer

ueq - Microequivalent

ug - Microgram

uS - Micro-Siemen

UV - Ultra-Violet

V/V - Concentration based on volume measurements

### APPENDIX A

# W & T:

W and T are low level data qualifiers assigned to data that are near or below the detection limit values (3)(5). The code <W indicates that no measurable response was observed under the test conditions. The reported value indicates the smallest amount that could have been measured under routine conditions. W is smaller than the standard deviation of duplicates near zero. The <T code is used to represent a measurable amount of the analyte which under the test conditions is not verifiable. The reported result should be used only for large batches of similar data to evaluate background levels or trends of contaminants in the environment where more sensitive analytical methods are not available.

To provide a consistent Laboratory Services Branch approach to data reporting, the Water Quality Section calculates W from the standard deviation of duplicates (S₂), near zero, by rounding down to the nearest 1,2 or 5 digit. T is five times W. The latest calculations, valid at date of publication for W and T values of all active work stations, are contained in this report. (APPENDIX B)

APPENDIX B
W AND T VALUES FOR DATA REPORTED IN 1990

PARAMETER	UNITS	WORKSTN CODE	TEST FULL CODE SCALE	W T
Acidity,	mg/L CaCO ₃	PHACD	ACDT 100 ACDG 1000	0.05 . 0.25 1.0 5.0
Gran	mg/L CaCO ₃	DOT RATS	ACDG 1000 ALKT 80 ALKT 1000	0.05 . 0.25 0.2 . 1.0
Fixed Endpoint 3.8.	mg/L CaCO ₃ mg/L CaCO ₃	WATS WQSDIRT	ALKT . 1000 ALKT . 1000 ALKT3 . 100	0.2 . 1.0 0.5 2.5 0.05 0.25
Gran	mg/L CaCO ₃	DOT RATS		0.05 . 0.25
Acid Ammonium Oxalate Extractable Citrate-Bicarbonate-Dithionite Extractable . Exchangeable Cation	% wt as Al meq/100g Al ug/L as Al	DOMETOX DOMETDI DOCATION . DOALSP	ALEOX 2	0.01 0.05 0.01 0.05 0.01 0.05 2 10 2 10
Sodium Pyrophosphate Extractable Soluble	% wt as Al ug/g as Al	DOMETALX . DOSOLAL . DOAAS	ALNDCV . 1000 ALEPY 0.5 . ALECA 40 ALUT 200	0.01 . 0.05 0.2 1 1 5
Total	*	DOAAS	CDUT 2	0.01 . 0.05 0.02 . 0.1
Exchangeable Cation	mg/L as Ca mg/L as Ca mg/L as Ca	PRAA400 PRAAS RMAAS WAAS DOCATION .	CAUR 2	0.02 . 0.1 0.02 . 0.1 0.1 . 0.5 0.2 . 1 0.01 . 0.05
Dissolved Inorganic	mg/L as C mg/L as C	DODIC	DIC 10 DIC 40	$\begin{smallmatrix}0.02&&0.1\\0.2&.&1\end{smallmatrix}$
Dissolved Organic  Total	<pre>% organic C % wt as C</pre>	ROM DOOXMAT DOTIC WAC	DOC 20 ORGC 40 TIC 2 TOC 50	0.1 0.5 0.01 . 0.05 0.01 . 0.05 1 5

W AND T VALUES FOR DATA REPORTED IN 1990

Fluoride

Hardness

Iron.

Lead,

#### UNITS WORKSTN TEST FULL W T PARAMETER CODE SCALE CODE Chloride 100 0.2 . . 1 . . . . . . . . . . . . . . . . mq/L as Cl . . . COCL CLIDUR CLIDUR 2 0.01 . 0.05 . . . . . . . . . . . . . . . . . . mg/L as Cl PRIC1 100 1 . . . 5 ug/filt Cl . . . PRLOV . . . CLIDUR 100 Chlorophyll, . . 50 0.2 . . 1 ug/L RCHLO CHLRAT CHLRAC . . 10 0.1 . . 0.5 "a" Acidified . . . . . . . . . . . . ug/L . . . . . . . RCHLO 10 "b" . . . . . . . . . . . . . . . ug/L . . . . . . RCHLO CHLRBT . . 5 · 0.1 . . 0.5 1 . . . 5 % wt as Clav . . DOPARTSZ CLAY 100 Colour DOCC COLTR . . 100 TCU 1 . WCOL COLTR . . 100 0.5 Conductivity 300 COND25 DOCC COND25 100 PRCON COND25 . 2000 RATS COND25 . 2000 WATS COND25 10000 WOSDIRT . . Copper, 0.2 . . 1

ug/L as F

Total . . . . . . . . . . . . . . . ug/L as Cu . . .

. . . . . . . . . . . . . . . mg/L as CaCO, . .

Acid Ammonium Oxalate Extractable . . . . % wt as Fe . . .

Citrate-Bicarbonate-Dithionite Extractable % wt as Fe . . .

Sodium Pyrophosphate Extractable . . . . . % wt as Fe . . .

. . . . . . . . . . . . . . . . . . mg/L as CaCO, . . . WAAS

. . . . . . . . . . . . . . . . . . mq/L as F

Acid Extractable . . . . . . . . . . . ug/g as Pb

Total . . . . . . . . . . . . . . ug/L as Pb

DOHMTE

DOSPF

WFNO3

RMAAS

DOASV . . .

DOMETOX . .

DOMETDI . .

DOMETALX .

DOHMTE . .

DOASV . . .

CUUT

CUUT

FFIDUR

FFIDUR

PBUT

PBUT

FEEOX . . .

FEEDI . . .

FEEPY . . .

. 50 . .

2

2

1

. . 70

8 9

HARDT . . . . . . .

HARDT . . . . . . . .

. . . 50

. . . 2

. .

0.3 . . 1.5

0.2 . . 1

0.2 . . 1

0.2 . . 1

. 0.05

. 0.05

0.01

0.01

0.01

APPENDIX B
W AND T VALUES FOR DATA REPORTED IN 1990

PARAMETER		UNITS	WORKSTN CODE	TEST FULL CODE SCALE	W I
		4			
Magnesium,					
*******		mg/L as Mg	PRAA400	MGUR 0.5 .	0.005 . 0.025
		mg/L as Mg	PRAAS	MGUR 2	0.005 . 0.025
		mg/L as $Mg$	RMAAS	MGUR 10	0.02 . 0.1
			WAAS	MGUR 50	0.1 0.5
Exchangeable Cation .		meq/100 g Mg	DOCATION .	MGESC 2.5 .	0.01 . 0.05
Manganese,					
Acid Ammonium Oxalate I			DOMETOX	MNEOX $0.1$ .	0.001 . 0.005
Citrate-Bicarbonate-Dit	thionite Extractable .	% wt as Mn	DOMETOX	MNEOX 0.1 .	0.001 . 0.005
Sodium Pyrophosphate Ex	xtractable	% wt as Mn	DOMETOX	MNEOX 0.05	0.001 . 0.005
Nickel,					
Acid Extractable	· · · · · · · · · · · ·	ug/g as Ni	DOHMTE	NIUT 50	0.2 1.0
Nitrogen,		150.5			
Ammonia plus Ammonium			DONUT	NNHTFR . 1000	1 5
		ug/filt N	PRAM	NNHTFR 50	0.05 . 0.25
	* * * * * * * * * * * *		PRAM	NNHTFR 2 '	0.002 . 0.01
				NNHTUR 2	0.002 . 0.01
		mg/L as N	RNDNP	NNHTFR 2	0.002 . 0.01
		mg/L as N	SDNP	NNHTFR 50	0.05 . 0.25
Nitrate		mg/L as N	PRIC1	NNO3UR 2	0.01 . 0.05
		ug/filt N	PRLOV	NNO3UR . 100	0.5 2.5
		ug/filt N	PRSEQ	NNO3FR 50	0.2 1.0
				NNRICF 50	0.2 1.0
Nitrate plus Nitrite .		ug/L as N	DONUT	NNOTFR . 1000	2 10
			RNDNP	NNOTFR 5	0.005 . 0.025
		mg/L as N	SDNP	NNOTFR 50	0.05 . 0.25
		mg/L as N	WFN03	NNOTUR 20	0.1 0.5
Nitrite			RNDNP	NNO2FR 0.2 .	0.001 . 0.005
			SDNP	NNO2FR 2	0.005 . 0.025
Total Kjeldahl			RTNP	NNTKUR 2	0.02 . 0.1
			STKNP	NNTKUR 50	0.05 . 0.25
Oxygen Demand,		■ ·★···································		*	
Biochemical		mq/L as O	SBBOD5	BOD5 400	0.2 1
Chemical			RCOD	COD 40	1 5
			SBCOD	COD 500	2 10

APPENDIX B
W AND T VALUES FOR DATA REPORTED IN 1990

PARAMETER	UNITS	WORKSTN CODE		FULL CALE	W	T
рЦ						
		DOCOP	РН	. 14		
		DOT		. 14		
		PHACD	PH	. 14		
*****		RATS	PH			
		WATS	PH			
* * * * * * * * * * * * * * * * * * * *		WQSDIRT	PH			
*************		DOSOILPH .	PHECA			
		DOSOILPH .	PHEW	14		
Phenolics,				2/2	40.00	.2
Reactive		MPHEN	PHNOL		0.2	1
Reactive	. ug/L Phenol	ROPHEN	PHNOL	. 50	0.2	1.
Phosphorus,		DARRE	DD04DD	100		
Bray II Extractable		DOBEP RNDNP	2	0.1 .	0.5	2.5 0.0025
마다 마다 보면 가장 다른 가는 가장 보면 가장 하는데 보다 되었다. 그래요 보다 가장 보면 가장 보면 보다는 것이 없는 데 없는 것이 없다면 없는 것이 없는 것이 없는 것이 없다면 없는 것이 없는 것이 없는 것이 없다면	54 1900 <b>244</b> Alia 1700 Million 18 18 18			A TOTAL STATE OF THE PERSON NAMED IN COLUMN NA		0.0025
Total			PPO4FR PPUT	0.2		0.01
100ai		CELLUID	PPUT	a contract to the		0.01
	. mg/LasP	DOP	PPUT1	100	0.02	1
	ing/pase		PPUT2	100	0.2	No.
Potassium,			LIUIZ	100	0.2	•
	mg/Las K	PRAA400	KKUR	1	0.005 .	0.025
		PRAAS	KKUR			0.05
		PRLOVAA	KKUR			1.25
		RMAAS	KKUR			0.05
		WAAS	KKUR		1501 (A) 180 (B) 180 (B) 180 (B)	0.25
Exchangeable Cation		DOCATION .		0.75		0.05
Sand					****	0.00
Silicon,	% wt as Sand	DOPARTSZ .	SAND	100	1	5
Acid Ammonium Oxalate Extractable	% wt as Si	DOMETOX	SIEOX	0.25	0.01 .	0.05
Reactive Silicates		ROM		10		0.25
Silt				- 7 .	****	V.120
	% as Silt	DOPARTSZ .	SILT	100	1	5
25 Mr. 9 CHO 199 MAR 42 24 MILLION MAR 40, WAI 5000 EM, EL 51 W 200 EM 100 MILLION MAR 500 MIL		AND OF CASE OF	- N		20 M W W 20	800

APPENDIX B
W AND T VALUES FOR DATA REPORTED IN 1990

PARAMETER	2.40 2.10 4.10	UNITS	WORKSTN CODE	TEST FULL CODE SCALE	W T
April 1997					
Sodium	75	The second second	SOUTH WILLIAM	999.202 ¥	0 005 0 005
		mg/L as Na		NAUR 1 .	
			PRAAS	NAUR 4	
		ug/filt Na	PRLOVAA	NAUR 50	
		mg/L as Na	RMAAS	NAUR 20	
		mg/L as Na	WAAS	NAUR 100 .	. 0.2 1
Solids, Dissolved		mg/L or mg/Kg .	SOLIDS	RSF 20000	. 2 10
				RSPA 3000	
Particulate		mg/L	SOLIDS	RSTA . 30000	. 0.5 2.5
		ug/filt as SO	PRSEO	SSO4FR . 250 .	. 1 5
				SSO4NF . 250 .	. 1 5
				SSO4UR 5 .	. 0.05 . 0.25
				SSO4UR 10 .	. 0.05 . 0.25
		ug/filt as SO, .	PRLOV	SSO4UR . 500 .	
		mg/L as SO,	RMDSO4	SSO4UR . 100 .	
Organo-Sulphur		mg/L as SO4	ORGSO4	SSO4UV 20 .	AND WAS INCOME.
Water Extractable Sulphur Dioxide				SSO4EW . 100 .	
		ug/filt as SO2 .	PRSEQ	SSO2FR . 350 .	. 1 5
Turbidity					
		FTU	RMTUB	TURB 200 .	
Zinc,				TURB 200 .	
Acid Extractable		ug/g as Zn	DOHMTE		. 0.5 2.5
Total		ug/g as Zn	DOASV	ZNUT 15 .	. 0.5 2.5

(7926) TD/380/P47/MOE